

METALLURGIA

The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

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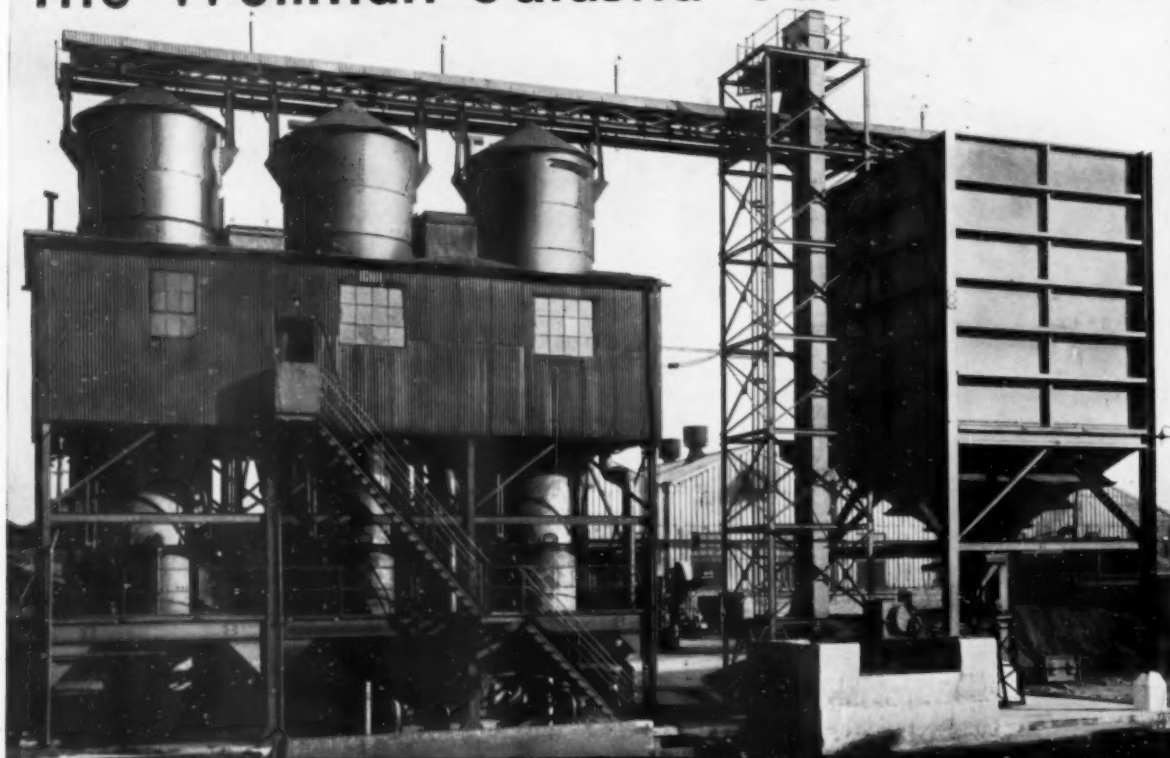
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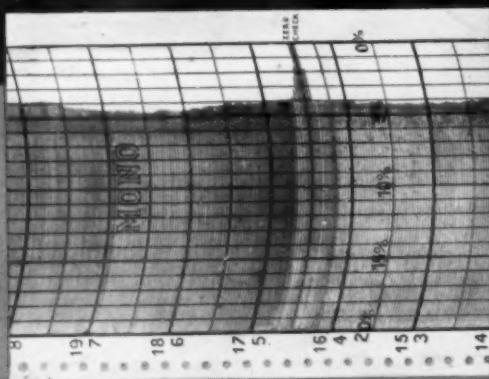
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METALLURGIA

THE BRITISH JOURNAL OF METALS

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The Problem of Materials for Marine Gas Turbines

THE evils of war have always been mitigated, to some extent, by spectacular advances in medicine and engineering and the last war was no exception. The development of the gas turbine, which made great progress, was made possible by metallurgical achievements of the past ten years and what must be regarded as the most outstanding of the achievements of the last war is the production of alloys capable of withstanding high stress at temperatures up to 750° C. These alloys were required for the aircraft jet engine, and since their first application invaluable data concerning their high temperature properties, particularly creep characteristics, have been accumulated.

The application of the gas turbine to marine propulsion was to be expected once suitable materials had been made available, but formidable problems, which had not arisen in the production of aircraft turbines, had to be considered. The most important factor in the adoption of the gas turbine for marine drives is the requirement of absolute reliability over a long life. In addition, particularly in merchant ships, such drives must operate economically, which depends on a rather sensitive balance between several factors, such as earnings from cargo carried, loading and discharging times and costs, running and capital charges, insurance, etc. Thus the reliability factor must be allied to an efficiency that compares favourably with other successful prime movers, when measured against fuel costs.

An aircraft turbine is expected to have a life of 300-400 hours, and under such conditions it is possible to conduct creep and other acceptance tests for periods at least as long as the service life of the engine; it is, therefore, possible to predict with considerable accuracy, the behaviour of the metals selected. The marine gas turbine, however, must be designed for a life of at least 100,000 hours and no data is available regarding the behaviour of metals when stressed for such long periods at elevated temperatures. Metallurgical stability must be assumed and allowable stresses can only be obtained by means of extrapolation as it is not practicable to await the results of 100,000 hour creep tests before using a specific material. If such a practice was adopted gas turbine development would be at a standstill and the metals would be out of date before they were used.

Stresses that can be used in gas turbines designed for 100,000 hours must be considerably lower than those used for 300-hour units and there is an urgent need for alloys that will carry higher stresses at yet higher temperatures. It is to be hoped, however, that the efforts of research workers will not be wasted in the development of new alloys which contain comparatively large percentages of rare elements. The continued supply of such alloys would be difficult if not impossible, should a large demand arise. That austenitic creep resisting

steels have a high coefficient of expansion together with a low thermal conductivity is well known. Production of alloys in which these two properties are brought nearer to those possessed by ferritic steels would be of considerable assistance to the designer.

A formidable problem facing the marine gas turbine at the present time is that of the corrosion of metals when in contact with certain types of fuel ash. The marine turbine must be capable of operating with a cheap fuel if it is to compete successfully with other established forms of propulsion. Distillate fuels such as gas and diesel oil are too expensive and the cheaper residual fuels must be used. Unfortunately most of these oils, particularly those of South American origin, contain vanadium, and it is the ash from these vanadium-bearing oils that has such a damaging effect on metals. The vanadium is present in the ash in the form of sodium vanadyl sulphate and produces an ash having a melting point in the region of 650° C. Marine gas turbines which are being developed at the present time have gas inlet temperatures of 650°-700° C., and all the ash produced in the combustion chamber is carried forward in a molten or sticky state and adheres to the turbine blades or air heater tubes, depending on whether the design is of the open or closed cycle.

It has been shown both with ash produced by the combustion of vanadium-bearing fuel oils and with synthetic ash consisting of V_2O_5 and Na_2SO_4 , that in its molten state the ash rapidly attacks all the well known gas turbine steels and alloys. The higher the temperature the greater is the rate of attack and its severity will be appreciated by the fact that some of these materials suffer a wastage of 25% at 670° C. and nearly 75% at 820° C. in a matter of 50 hours. Investigations into the possibilities of protective coatings have so far proved disappointing, but work is still in progress in this direction. Consideration is also being given to methods of collecting the molten ash particles by mechanical means at some point between the combustion chamber and the turbine inlet.

Should a metal or protective coating be found that will give complete immunity against the corrosive effects of vanadium-bearing fuel ash there would still remain the problem of fouling. Owing to the sticky nature of the ash it does not pass through the turbine but accumulates on the blades and results in a progressive lowering of efficiency.

The development of turbines having gas temperatures of 1,000° C. and above, with blades maintained below the melting point of the ash by means of liquid cooling will almost certainly result in the elimination of corrosion and may possibly reduce blade-fouling.

On a recent visit to Pametrada, reference to the activities of which is made elsewhere in this issue, it was found that this Association was very much alive to these problems and that active research both on a laboratory scale and with full scale commercial sized equipment was in progress.

Price Stability in Key Industries

APART from the need for improved efficiency in industry the most essential requirement to enable the exporting industries to compete in the world's markets is the price stability of what can be regarded as their raw materials. With the exception of aluminium, the prices of non-ferrous metals are continually rising, and to such an extent that the maintenance of stable prices for products manufactured from them is impossible, and variations, which sometimes take effect from day to day, make quotations for the supply of products a nightmare to progressive firms. Can it be said that bulk buying by the Government has been beneficial to industry? Would it not be more profitable for the Government to announce measures to maintain stability of prices and combat inflationary tendencies in a manner consistent with its desire to maintain full employment.

The maintenance of full employment is undoubtedly a major objective, but it depends largely on overseas trade and for success prices are as important as quality and they cannot be maintained at their present high level unless successful efforts are made to prevent persistent rising costs. Some time ago railway freight charges were substantially increased and shortly afterwards an estimate was given in Parliament that the National Coal Board, as a result of these increased rates would have to increase its prices by an average of 1s. 6d. a ton for industrial and 2s. 6d. a ton for domestic fuel. Since then a rise in the cost of petrol and fuel oil has further complicated the position, making road transport charges excessive, and seriously interfering with those industries trying to reduce costs of products destined to compete with the products of countries overseas.

It is particularly fortunate that the Iron and Steel Federation has announced that, for the present at any rate, the steel industry will absorb its increases in costs, amounting to about 10s. a ton, without asking for an increase in prices. This is made possible largely by the industry's record of production and productivity, in other words by improved efficiency. One result of increased production will be that fewer steel imports will be needed—the cut is to be from 500,000 tons to 200,000 tons of semi-finished steel; and this, together with a switch to the cheaper Continental sources of supply, will save £3.3 million. The second result is that fixed and other costs will be spread over a larger tonnage, and this will save £1.5 million. But just over £1 million still remains which will have to come from the industry's profits.

This contrast between the behaviour of nationalised industries and the unnationalised iron and steel industry will not have passed unnoticed. So put it no higher, as stated in *The Financial Times*, it is an illustration of the usefulness to the community of an industry that makes profits. It should be noted that the steel industry, since it last put up its prices, has absorbed increases in the costs of its principal raw materials of something of the order of 7s. 6d. a ton. Although as an object lesson this is valid and valuable, it will be appreciated that the National Coal Board is still enjoying a seller's market—indeed the country's stocks of coal are being drawn upon to satisfy demand—while the steel industry is moving into the buyer's market. This generalisation does not hold good in detail either for all types of coal or for all types of steel. Some types of coal are in surplus supply on the European market, while some types of steel, such as sheets and tinplate, are still in short supply in this

country. The steel industry, therefore, is facing a weakening market and farther away in the future there is the possibility of a European surplus of some eight million tons.

The remarkable thing is that in all nationalised industries, as well as those in which the Government has made itself responsible for raw materials, rising prices are hampering many industries in their efforts to develop overseas trade. There is a need for the maintenance of a general policy by the Government, with regard to these key industries and materials, that must not be allowed to remove the spur to efficiency, and measures to effect stability of prices would be invaluable.

Standardisation

THE term standardisation covers a very wide field, but still is confined to one general idea, namely, the establishment of an agreed basis to serve as a reference for the assessment of the suitability of a product. It takes two main forms which are usually described as functional and dimensional. The former is the establishment of the means of expressing quantitatively or qualitatively the fitness for purpose of materials, articles, components, machines, structures, etc., where the Imperial standards of weights and measures do not adequately apply. Dimensional standardisation is designed to achieve simplification, unification and interchangeability. It may also be used as an indirect method of specifying quality.

In functional standardisation standards have taken various forms, such as, terms and definitions, quality and performance, methods of test, and codes of practice, all designed to secure uniformity and accuracy of description, to provide the measure whereby fitness for purpose can be expressed, to ensure uniformity in the definition and properties by diminishing variations in test results due to differences in test procedure, to define and guide the correct application of materials, etc., and thus secure the adequacy of the result.

Although the need for the use of well defined standards was never greater than to-day this does not mean that their application is so simple that they can be translated in practice by anyone, indeed considerable experience and ability is frequently necessary to overcome difficulties that arise through slavish adherence to the interpretation of a particular standard. Several of these are cited in a controversial article which appears elsewhere in this issue. With many materials, upon which work is to be carried out by the purchaser, a more satisfactory approach to acquiring a yard-stick is in close co-operation with all parties concerned in the transaction, especially the purchaser and manufacturer. It is preferable to submit samples which can be submitted to the actual operations involved in producing the finished articles, but adequate guidance should be given on the work to be performed on the sample so that it may fulfil its ultimate service purpose.

The author of the article referred to has emphasised the case of cold-rolled non-ferrous materials, but much of his comment is applicable in a wider sense. The subject is a contentious one but there can be no doubt that, with a proper interpretation of the particular purpose for which the material is to be used and the fabricating technique to be employed, there would be less difficulty in supplying a material to a standard satisfactory to all concerned.

Some Properties of Tantalum-rich Alloys with Wolfram and Molybdenum

By Rupert H. Myers, M.Sc., Ph.D.*

In a study of tantalum-rich alloys with wolfram and molybdenum, it has been established that more than 10 atomic per cent. of the added metals render tantalum very difficult to work at room temperature. Considerable increases in strength are obtained, but the ductility is lowered. Molybdenum additions to tantalum raise the electrical resistivity much more than wolfram. The presence of up to 10 atomic per cent. of wolfram or molybdenum in tantalum raises the annealing temperature by a maximum of 50° C.

FROM a study of the interatomic distances of the three metals concerned—namely, tantalum, wolfram and molybdenum, together with a consideration of valency effects involved, it appeared very likely that tantalum would form wide series of solid solutions with both wolfram and molybdenum and also that it was unlikely that any compounds would be formed. These theoretical predictions are borne out by the small amount of experimental work reported in the literature.¹ Because of the scant knowledge of the properties of tantalum alloys with the high melting point metals it was proposed, as a first step, to investigate those alloys with wolfram and molybdenum which are workable at room temperature. It is well known that wolfram and molybdenum wires are very brittle in the annealed condition, whereas tantalum is extremely ductile, providing it has been annealed in a high vacuum. However, wolfram and molybdenum are much harder and stronger than tantalum and it was thought that the latter could be strengthened by alloying with them. This would have distinct advantages if the metal were to be used as filaments, as high-temperature sagging and room temperature brittleness could be overcome.

Preparation of the Alloys

Materials used and method of mixing.—Electrolytic tantalum powder and wolfram and molybdenum powders produced by hydrogen reduction of wolfric oxide and ammonium molybdate, respectively, were used for the preparation of the alloys. All metals were of a purity 99.95% or better. Qualitative spectrographic analyses indicated the following impurities:—

WOLFRAM	TANTALUM	MOLYBDENUM
Mo Nil	Ni Trace	Cu Trace
Fe Faint trace	Fe "	Ni Faint trace
Cu "	W Faint trace	Fe " "
Ca " "	Cu " "	W Nil " "
Si Trace	Ca " "	
Mg Nil	Si " "	
Al "	Cb Nil	
Ti "	Pb "	
Cr "	Sn "	
Ni "	Cr "	
Co "		

Chemical analysis was employed to check several impurities with the following results:—

WOLFRAM	TANTALUM	MOLYBDENUM
SiO ₂ 0.02%	Ni 0.01%	Cu 0.013%
	Fe 0.02%	

Just prior to their use the wolfram and molybdenum powders were heated to 900° C. for 30 minutes in a stream of pure dry hydrogen and then degassed by heating to 1,000° C. in a vacuum of 10⁻⁴ mm. Hg. pressure. This was to prevent the possible expansion of pressed compacts during sintering due to the too rapid release of gas; it was known that the gas content of the tantalum was such as to cause no trouble in this respect. Since the alloys were not to be fused it was considered imperative to obtain as intimate a mixing as possible of the metal powders prior to pressing, because any inhomogeneities in the bars after this stage would have to be removed by the relatively slow process of diffusion in the solid state. The powders were screened separately through a 150-mesh Tyler sieve and the over-size rejected. Then the required weights of powders, sufficient for one bar at a time and of a definite composition, were weighed out and placed on a sheet of clean paper. The powders were rolled together for several minutes and then passed again through the 150-mesh sieve. This served to break down the aggregates of wolfram or molybdenum powder which tended to form and enabled the particles of the two powders to come into intimate contact. The mixture was rolled again for 10 minutes and placed in a glass tube. Just prior to pressing, the powder was re-rolled and loaded into the die as uniformly as possible. A pressure of 50 tons/sq. in. was used throughout this work.

Bars 5 in. long with $\frac{1}{4}$ in. square cross-section and containing approximately 1, 5, 10 and 20% (atomic) of wolfram and molybdenum in tantalum were made in this way.

Consolidation of the pressed bars.—After pressing, the bars were mounted individually in the grips of the vacuum sintering furnace² and when the system was evacuated to a pressure of 10⁻⁴ mm. Hg. the current was turned on. The heating schedule was the same as that for the sintering of tantalum,³ and involved a final heating for 4 hours at 2,600° C. Rather than employ prolonged heating at this stage it was thought better to subject the bar to a certain amount of deformation and then to reheat it since this, especially if repeated several times, would provide good conditions for the attainment of a homogeneous alloy. Where possible then, the heating and working schedule given in Table I was followed.

* Formerly of the Baillieu Laboratory, University of Melbourne, now a member of the Australian Commonwealth Scientific and Industrial Research Organisation's team at the Atomic Energy Research Establishment, Harwell, Berkshire.

1 See Myers, R. H., *Metallurgia*, 1948, **39**, 57, for a summary of published data on tantalum alloys.

2 *Ibid.*, *Proc. Aust. I.M. & M.*, 1946, **144**, 45.

3 *Ibid.*, *Metallurgia*, 1948, **38**, 307.

Certain of the alloys were too brittle to withstand the swaging and they were not subjected to the reheating treatments. The compositions of the alloys which were prepared are given in Table II, the methods developed for the analytical determination of wolfram and molybdenum in tantalum being given in the Appendix.

TABLE I.—HEATING AND WORKING SCHEDULE FOR PREPARATION OF ALLOY RODS

Time at 2,600° C. (hours)	Subsequent deformation by swaging—Reduction in area—%
Sintering .. 4.0	40
Reheating .. 1.5	25
" .. 0.5	20
" .. 1.0	48
" .. 0.5	(0.1 in. Stock)
Total .. 7.5	

From Table II it will be seen that there was a small but general increase in the percentage of wolfram and decrease in the percentage of molybdenum in the alloys after the heat treatment. This would seem to indicate that the relative volatilities of the three metals at 2,600° C. are:—

molybdenum \gg tantalum $>$ wolfram.

This is in the same order as the melting and boiling points.

TABLE II.—COMPOSITION OF THE ALLOYS

Alloy	As prepared by weighing		By analysis after final heating	
	Weight %	Atomic %	Weight %	* Atomic %
Wolfram				
ABY	1.0	0.99	1.20	1.18
ABZ	5.0	4.92	5.20	5.12
ACA	10.0	9.85	10.40	10.25
ACB*	20.0	19.8	20.4	20.2
Molybdenum				
AFL	0.50	0.87	0.40	0.78
AFM	2.50	4.61	2.2	4.2
AFN	5.50	9.90	5.20	9.6
AFO*	11.7	20.0	11.2	19.7

* Chemical analysis of these alloys was performed on the "as-sintered" material.

The swaged and annealed alloy rods of 0.1 in. diameter were used as stock material and any further deformation required was achieved by wire drawing. The technique used was the same as that which has been described for drawing tantalum,⁴ but extreme care had to be taken to ensure the complete covering of the wire with the anodically applied film and with the lubricant, as there was a marked tendency for the harder wires to score due to pick up on the die. The smallest wire drawn was 0.009 in., this being the limit of the dies available at the time.

Some Properties of the Alloys

General.—As has been mentioned earlier it was proposed to produce rod of about 0.1 in. diameter for use in determining the properties of the alloys. However, it was found that alloys ACB* and AFO* could not be swaged at room temperature without extensive cracking. Heating to about 500° C. did not improve the workability; higher temperatures were not used because of the oxidation of the hot alloys in air. It appeared that the limit of alloying additions of wolfram and molybdenum to tantalum, without destroying the cold workability of the latter, was of the order of 10%

⁴ *Ibid.*, 1948, 30, 7.

* These letters apply to alloys with compositions as listed in Table II.

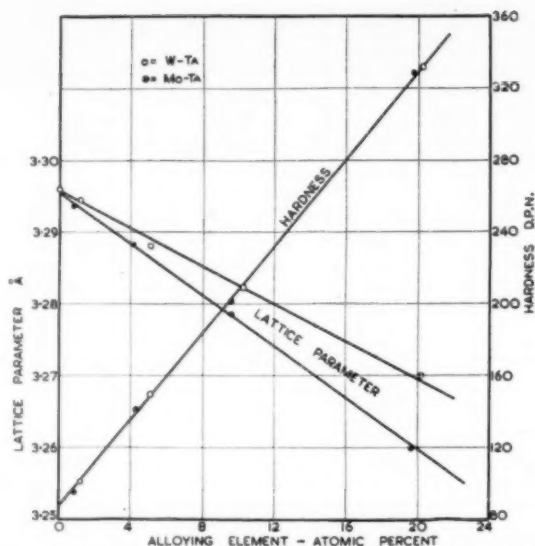


Fig. 1.—Effect of additions of wolfram and molybdenum on the lattice parameter and hardness of annealed tantalum.

(atomic). Actually, occasional cracks were found in alloys of this composition so that they must have been just on the borderline.

All of the alloys were examined microscopically and appeared to be homogeneous. The etchant used was cold concentrated nitric acid containing 5% concentrated hydrofluoric acid. The etchant was swabbed on to the specimens which were mounted in Lucite, this being more chemically resistant than bakelite.

Lattice parameter.—X-ray back reflection photographs were taken of each of the alloys and, from these, lattice parameters were determined. In all cases except the 20% (atomic) alloys, metallographically polished, annealed specimens of 0.1 in. diameter were used for the photographs. The approximately 1, 5 and 10% (atomic) alloys appeared to be homogeneous, but alloys AFO and ACB, which were used in the sintered condition showed that complete solution had not quite been obtained. The results of the lattice parameter determinations are plotted as a function of composition in Fig. 1. There was no evidence of intermetallic compounds in any of the alloys.

Electrical resistivity.—The effect of additions of wolfram and molybdenum on the electrical resistivity of tantalum was studied, using annealed alloy rods of 0.1 in. diameter. The results are plotted as a function of composition in Fig. 2, the curves obtained being typical of series of solid solutions. It will be noted that small amounts of alloying elements have quite an appreciable effect on the electrical resistivity of tantalum. Molybdenum had a much greater effect than wolfram and on a weight for weight basis this would be even more marked than is evident from Fig. 2. The percentage increases in resistivity for two alloys of each group are given in Table III.

Ultimate tensile strength, elongation and hardness.—These properties were determined on the workable alloys of tantalum with wolfram and molybdenum using 0.1 in. diameter swaged rod which had been annealed at 2,600° C. for 30 minutes in a vacuum of 10^{-4} mm. Hg pressure. The elongation was measured

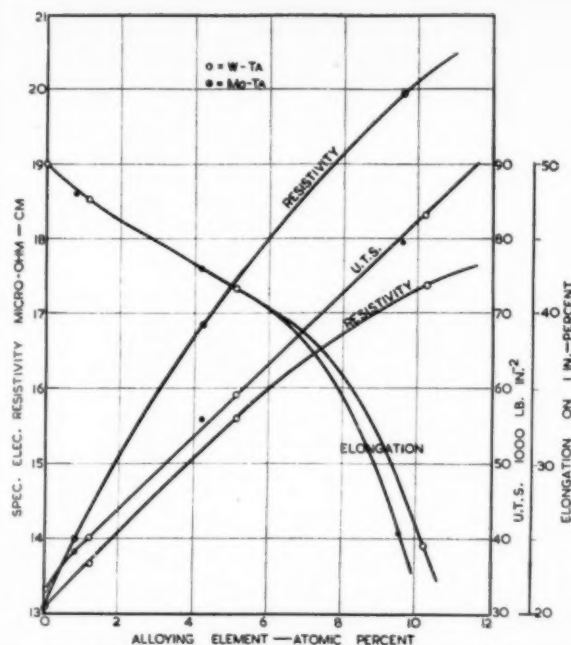


Fig. 2.—Effect of additions of wolfram and molybdenum on the electrical resistivity (20° C.), ultimate tensile strength and elongation of annealed tantalum.

on a gauge length of 1 in. and the surface for the hardness determinations was obtained by mounting a short piece of rod about $\frac{1}{2}$ in. long in Lucite and polishing it metallographically to give a diametrical longitudinal section. In the case of alloys AFO and ACB the hardness was determined on a polished section of the "as-sintered" material. Because of the slight porosity of the sintered alloys a large number of measurements was made and only the highest values used.

The mean values obtained in this series of experiments are plotted as a function of composition in Figs. 1 and 2. It will be seen that approximately linear relationships hold for the hardness and ultimate tensile strength of the alloy series. Marked increases occurred when either wolfram or molybdenum was added to tantalum. For example, 10% (atomic) of wolfram or molybdenum increased the U.T.S. of tantalum by a factor of $2\frac{1}{2}$ and commensurate increases in hardness were obtained. It was interesting to note that equal atomic additions of wolfram or molybdenum caused changes of the same order. This was not surprising in view of the close similarity of their atomic diameters.

GOLDSCHMIDT'S ATOMIC DIAMETER

Tantalum	2.94
Wolfram	2.82
Molybdenum ..	2.80

It will also be noticed from Fig. 2 that additions of 10% (atomic) wolfram or molybdenum to tantalum caused marked decreases in elongation. The values obtained, however, were still greater than

TABLE III.—PERCENTAGE INCREASE IN ELECTRICAL RESISTIVITY WHEN WOLFRAM AND MOLYBDENUM ARE ALLOYED WITH TANTALUM

Alloy composition		Increase in resistivity at 20° C.—%
Atomic %	Weight %	
W in Ta		
1	1.01	3.7
10	10.15	32.0
Mo in Ta		
1	0.53	8.4
10	5.56	53.5

for pure wolfram or molybdenum. (See Table IV).

Resistance to oxidation.—All of the alloys prepared—i.e., up to 20% (atomic) of wolfram and molybdenum in tantalum—were tested to determine the temperature at which a non-adherent oxide was formed. There appeared to be no difference between the alloys and pure tantalum, temperatures above about 620°C., in air, being sufficient to form the non-adherent film.

Effect of Deformation on Mechanical Properties

A series of experiments was performed to determine the effect of deformation on the mechanical properties of the workable alloys of tantalum with wolfram and molybdenum, the deformation being produced by wire drawing as described earlier. Hardness, ultimate tensile strength and elongation were determined on wires which had been subjected to varying amounts of deformation and the mean values obtained are plotted in Figs. 3-8.

TABLE IV.—MECHANICAL PROPERTIES OF ANNEALED TANTALUM, WOLFRAM AND MOLYBDENUM

Metal	U.T.S. lb./sq. in.	Elongation % (1 in.)	Hardness D.P.N.
Tantalum	33,500	50	80-90
Wolfram	150,000*	0*	350
Molybdenum ..	171,000	10-25	140-185

* These values are in doubt owing to the brittle nature of fully annealed wolfram at room temperature.

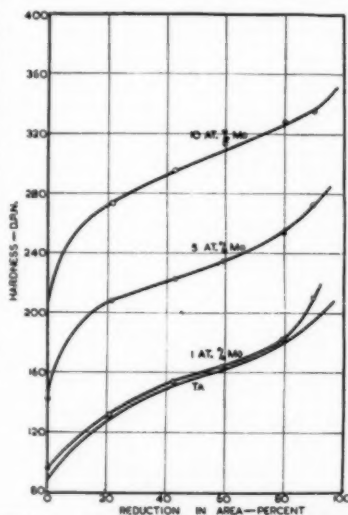


Fig. 3.—Effect of cold deformation on the hardness of tantalum-rich alloys with molybdenum.

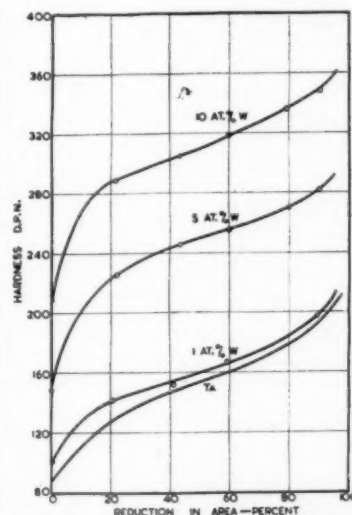


Fig. 4.—Effect of cold deformation on the hardness of tantalum-rich alloys with wolfram.

Note.—The compositions given in these and subsequent figures are nominal only, and reference should be made to Table II for the exact analyses.

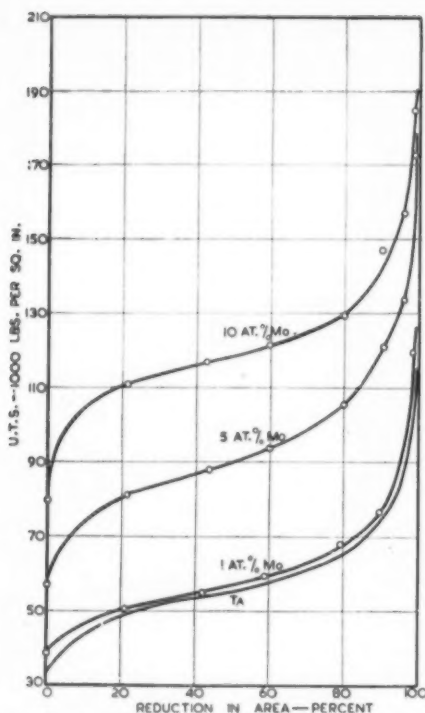


Fig. 5.—Effect of cold deformation on the ultimate tensile strength of tantalum-rich alloys with molybdenum.

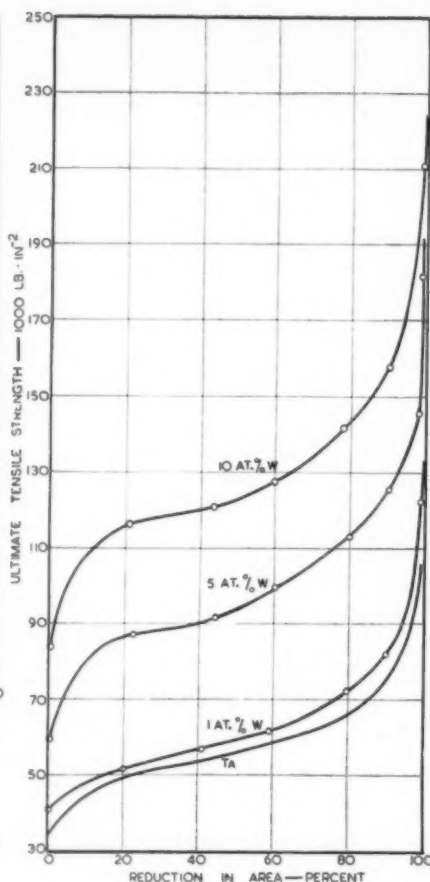


Fig. 6.—Effect of cold deformation on the ultimate tensile strength of tantalum-rich alloys with wolfram.

From the hardness and U.T.S. curves it was evident that the 5 and 10% (atomic) alloys work-hardened more rapidly than the pure tantalum or 1% (atomic) alloys. Tantalum is exceptional in that it work-hardens much more slowly than most metals, especially those in the high melting point group, and it is fortunate that it can be worked at room temperature because of the rapid deterioration it would suffer if working temperatures as high as those needed for wolfram and molybdenum had to be employed.

Small amounts of deformation had a large effect on the elongation as can be seen from Figs. 7 and 8.

The maximum tensile strength recorded for these alloys was 210,000 lb./sq. in. for approximately 10% (atomic) wolfram in tantalum after a deformation of 99% reduction in area. The

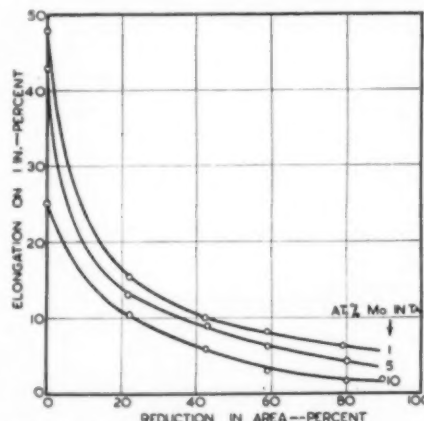


Fig. 7.—Effect of cold deformation on the elongation of tantalum-rich alloys with molybdenum.

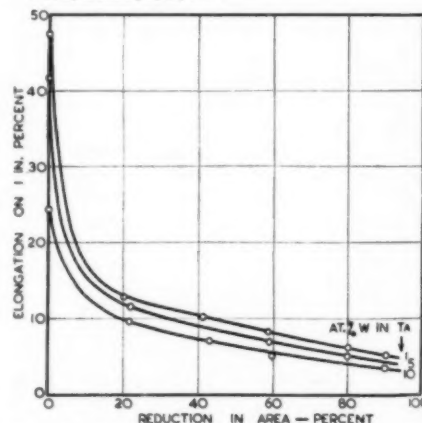


Fig. 8.—Effect of cold deformation on the elongation of tantalum-rich alloys with wolfram.

diameter at this stage was 0.009 in. The close relationship between the effects of wolfram and molybdenum has manifested itself throughout the work, additions of wolfram giving ultimate tensile strength, elongation and hardness values practically identical with those for equal atomic additions of molybdenum.

Annealing Temperature of Some Tantalum-rich Alloys with Wolfram and Molybdenum

General.—The effect of deformation on the annealing temperature of tantalum had been investigated earlier in some detail, and it was decided to study the effects of alloying wolfram and molybdenum with tantalum. In the earlier work it was found that tantalum, at elevated temperatures, was an exceedingly powerful absorber of gases and that this absorption caused very considerable increases in hardness⁵. Before any systematic work was done on the alloys it was decided to investigate whether this ability to absorb gases was in any way altered by the addition of wolfram and molybdenum in quantities up to 20% (atomic).

It had been found that the only way to prevent tantalum specimens from absorbing gases at approximately 1,800° C. (the temperature at which maximum hardening occurred) was to enclose the specimens in a tantalum tube and to heat them in as high a vacuum as possible. The tantalum tube acted as a barrier to the gases present in the system, which was at a pressure of

⁵ *Ibid.*, *Metallurgia*, **41**, 301.

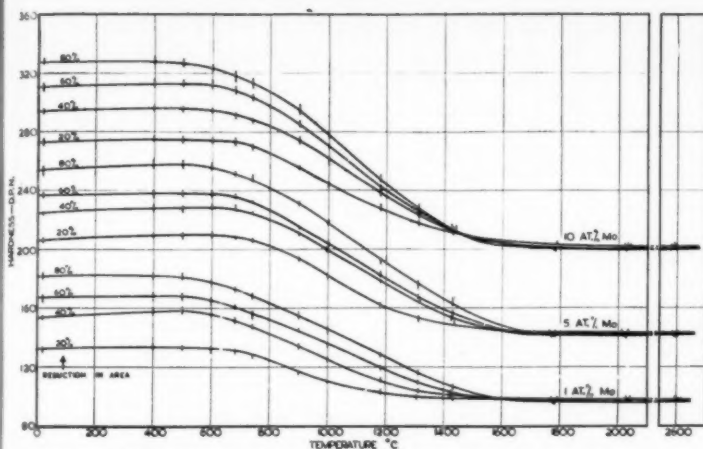


Fig. 9.—Hardness of deformed tantalum-molybdenum alloys after heating at various temperatures in vacuo for 30 minutes.

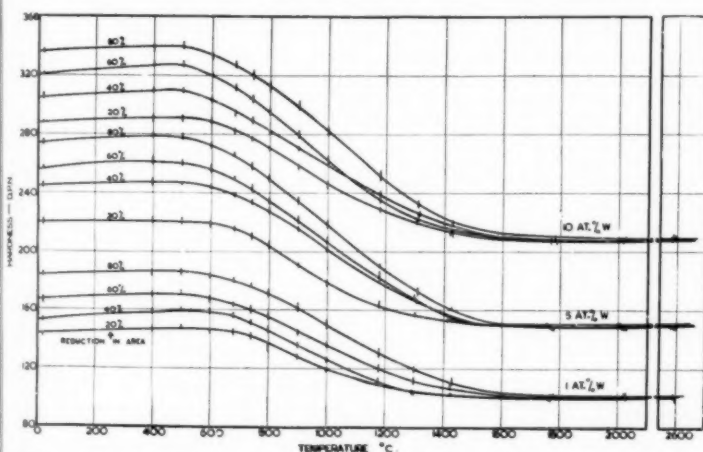


Fig. 10.—Hardness of deformed tantalum-wolfram alloys after heating at various temperatures in vacuo for 30 minutes.

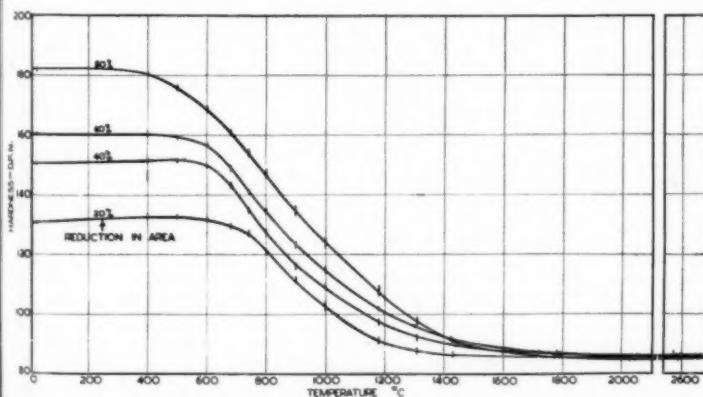


Fig. 11.—Hardness of deformed tantalum after heating at various temperatures in vacuo for 30 minutes.

not more than 10^{-4} mm. Hg., and tended to absorb the gases which would have otherwise gone into the specimens. The tube gradually became hard. In this case heating was by high-frequency induction.

tube by means of an optical pyrometer. The emissivity of tantalum was taken as 0.35 and the appropriate corrections were made.

Results.—The hardness measurements obtained are

When unprotected specimens were heated by induction by being suspended in a coil, they usually showed an increase in hardness. It was decided, then, to heat specimens of the unprotected alloys to 1800°C . by induction; they all exhibited the hardening phenomenon. When specimens were protected in a tantalum tube and then heated by induction no hardening occurred. As a result of this preliminary work on the alloys it was decided to retain all the precautions which have been described earlier in the work on the annealing of pure tantalum.⁵

The general plan was to determine, by means of hardness measurements, the annealing temperatures of various tantalum alloys which had suffered varying amounts of cold work and to study the effect of composition and deformation on the annealing temperature.

Procedure.—Fully annealed rods, 0.1 in. diameter, of each of the six workable alloys of tantalum with wolfram and molybdenum were deformed by wire drawing to give wire specimens which had suffered approximately 20, 40, 60 and 80% reduction in area. The anodic film on the wires was removed after drawing by polishing with fine emery cloth because it was desired to exclude all possible sources of contamination. Chemical cleaning was not used because of the possibility of hydrogen being absorbed by the metal. Short lengths of the wires were mounted and polished metallographically to the diametrical longitudinal section, and hardness measurements were made on the flat faces along the axes of the specimens. Each series of specimens was heated for 30 minutes at various temperatures in a vacuum of 10^{-4} mm. Hg. pressure, obtained by the use of rotary and oil diffusion pumps. In the case of the lower temperatures the specimens were sealed in small molybdenum cylinders and heated in a vitreous silica tube, the highest temperature used with this method being 900°C . At the end of the 30 minutes heating the furnace was removed from the silica tube and the specimens cooled from the highest temperature used to below 100°C . in about 20 minutes. Higher temperatures were obtained using high-frequency induction heating and the specimens were heated in a closed tantalum cylinder in the work coil. In this case the specimens cooled to below red heat in approximately 45 seconds.

The temperature in the silica tube was measured with a Chromel-Alumel thermocouple and in the tantalum

shown plotted as a function of temperature in Figs. 9 and 10, the vertical lines in the graphs representing the scatter of the hardness values. The approximate deformation, as reduction in area-%, is marked on each curve. For the purpose of comparison the hardness-temperature curves determined for pure tantalum are reproduced in Fig. 11. It will be seen from the curves that the presence of up to 10% (atomic) wolfram or molybdenum in tantalum increased only slightly the annealing temperature; the maximum increase amounted to approximately 50° C., this maximum occurring with the 10% (atomic) alloys. The hardness of the alloys began to drop between 500° and 700° C. depending on the amount of deformation prior to heating. Those alloys which had been deformed by 20% began to soften at about 700° C. while alloys which had suffered 80% reduction in area began to soften just above 500° C. The softest state appeared to be reached with all alloys at a maximum of about 1,600° C., the more heavily deformed specimens being the last to reach the dead soft condition.

It was interesting to note that the heating of the alloys *in vacuo* caused the surface of the specimens to acquire an etched appearance. This was first noticed in the alloys which had been heated to about 1,750° C. and the effect was only detectable under the microscope. After heating to higher temperatures the vacuum etching became appreciable and was easily seen with the unaided eye.

Summary

A study of the tantalum-rich alloys with wolfram and molybdenum has been made and the following points established:

1. Addition of more than 10% (atomic) wolfram and molybdenum makes tantalum very difficult to work at room temperature; heating to 500° C. does not improve the workability.
2. The presence of wolfram and molybdenum in tantalum increases the ultimate tensile strength, hardness, resistivity and work-hardening rate, and decreases the elongation.
3. The resistance to oxidation of the alloys is no better than tantalum, temperatures above about 620° C. being sufficient to form a non-adherent oxide film.
4. The presence of up to 10% (atomic) wolfram and molybdenum in tantalum raises the annealing temperature by a maximum of 50° C.
5. The alloys are as susceptible to gas hardening at elevated temperatures as is tantalum.
6. Equal atomic additions of wolfram and molybdenum cause the same order of changes in mechanical properties, although molybdenum has a much greater effect on the resistivity.

In general, small additions of wolfram and molybdenum strengthen tantalum without destroying its cold workability and the alloys are not brittle in the annealed state. Because of these properties the alloys may be useful for filaments and filament supports where rigidity and resistance to mechanical shocks are required.

Appendix

Chemical Analysis of Alloys

From a study of Table II it will be seen that chemical analysis of the final consolidated alloys was necessary in view of the change in the compositions during heat-treatment. No reference could be found in the literature

to methods of analysing alloys rich in tantalum, and it was necessary to develop new, and modify existing analytical procedures for treating the consolidated metals. This was done in collaboration with Miss R. Shoebridge* and Mr. B. D. Guerin†, who kindly performed the analyses.

In the case of tantalum-rich alloys with wolfram and molybdenum there appeared to be two methods for obtaining the alloys in solution:—

- (a) Oxidation of the alloy by heating and subsequent fusion of the oxides with potassium pyro-sulphate or potassium carbonate.
- (b) Solution of the alloys in acids.

The only acid which could be used for tantalum alloys was hydrofluoric and this usually interfered with most analytical procedures so that a combination of the two methods had to be used. It was thought inadvisable to convert the alloys to oxides by heating in air, owing to the high local temperatures which developed when tantalum was oxidized. These would have caused portion of the oxides of wolfram and molybdenum, which are volatile, to be lost, thus giving erroneous results.

The methods finally adopted were checked using weighed amounts of the pure powders and were found to give accurate and reproducible results.

Determination of wolfram in tantalum.—The sample was dissolved in hydrofluoric acid plus nitric acid and a small amount of sulphuric acid was added and the solution was then taken to dryness. The dried mass was broken up, mixed with potassium carbonate and fused. The procedure from this point was as described by Schoeller and Powell⁶ for the determination of wolfram in tantalum ores. It involved the separation of magnesium tantalate from the potassium wolfrate and tantalate solution by the addition of magnesium sulphate. The wolfric acid was recovered using the tannin-cinchonine method. The weight of sample used for each determination was adjusted so that the wolfric oxide present did not exceed 0.02 g., since larger amounts than this necessitated a double separation.

Determination of molybdenum in tantalum.—The sample of alloy (0.25 g.) was digested with nitric acid in a tall crucible, and hydrofluoric acid added slowly. When the alloy had dissolved completely, several drops of sulphuric acid were added and the solution evaporated to fumes or dryness. Fusion with 3 g. potassium bisulphate was carried out until a clear melt was obtained. This was lixiviated with hot 10% tartaric acid (30 ml.) and then diluted to 300 ml. The solution was made distinctly ammoniacal, heated almost to boiling and hydrogen sulphide was passed through the solution for 15 minutes. Sulphuric acid was added to make the solution distinctly acid and hydrogen sulphide was passed through the heated solution (80° C.) for a further 5 minutes. After coagulation of the precipitated molybdenum sulphide, it was collected on a No. 40 Whatman paper. The precipitate was washed well with hydrogen sulphide water containing hydrochloric (not sulphuric) acid. From this stage the procedure was as usually adopted for the determination of molybdenum as lead molybdate (PbMoO₄, containing 26.13% Mo). It involved the solution of the sulphide in 5% potassium hydroxide and its subsequent oxidation

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† School of Metallurgy, University of Melbourne.

⁶ Schoeller, W. R., and Powell, A. R., "The analysis of minerals and ores of the rarer metals." (Griffin & Co., 1940).

using bromine water. Lead molybdate was precipitated by adding, dropwise, 4% lead acetate in 1% acetic acid to the solution which contained 5 ml. glacial acetic acid and 12 g. ammonium acetate. The precipitate was collected and ignited at a dull red heat for 10 minutes.

Acknowledgment

This work was performed in the Baillieu Laboratory, University of Melbourne, Australia, under the general direction of Professor J. Neill Greenwood, whose counsel the author very gratefully acknowledges.

The Use of Alloying Elements in Chilled Iron Rolls

By Archibald Allison

*Author of "The Manufacture of Chilled Iron Rolls"
Formerly Chief Metallurgist of J. J. Habershon & Sons Ltd.*

Chilled cast iron rolls, even of the straight-carbon type, are delicate and highly strung castings, and few there are who are bold enough to guarantee any to give service until worn out. With alloyed rolls the highly strung characteristics are even more emphasised. The work they are required to perform is very severe, and it is largely rough treatment that is responsible for the casualties among rolls. In this article are discussed some aspects of the manufacture of chilled alloy cast-iron rolls, illustrated by reference to particular practices.

THE application of alloying elements to a sensitive material like chilled iron calls for careful consideration and great caution. Since the depth of chill is itself a resultant of many factors the effect of each alloy addition must be balanced with other effects.

With the object of increasing hardness, and therefore resistance to abrasion, chromium is the simplest natural addition, and since chromium has a stabilising effect in the carbide approximately equal to the instability given by silicon, small additions of chromium must be balanced by similar increases of silicon content.

It now appears probable that the dendritic crystals in the microstructure are not pure carbide but complex carbides, in the decomposition of which silicon plays an important part, limited by the time effect in cooling produced by the balance of the quenching action of the chill mould and the residual heat of the interior metal. The time effect is marked by the extended zone between chill and grain, when alloys are used, marking the slower decomposition of the primary carbides.

The effect of sulphur in stabilising the carbide to produce white iron has long been known, but recent research indicates that the effect is due to the complexity of the primary carbide. Chromium up to 0.4% may be used to give slightly greater hardness by 3 or 4 points on the scleroscope scale without disadvantage.

The addition of nickel to iron and steel is characterised by sluggish reaction in the phase changes producing the phenomenon known as lag. In cast iron generally, nickel has the effect of producing a finer structure in the pearlite and hence a stronger metal. The effect upon the chill is to produce a less stable carbide like silicon, and hence like silicon to reduce chill depth, but only to the extent of approximately one-third the effect of silicon. The addition of nickel, therefore, has to be balanced by lowering the silicon, or by adding chromium. The maximum amounts added are usually 4.5% nickel and 1.5% chromium. This rule of three to one with alloys is also useful when employing lower percentages, and there is much scope for varying the amounts according to size and purpose of the rolls.

Since molybdenum has been found useful in alloy

steels there is reason to think that a certain percentage would be a valuable addition to chilled iron if cost permits. On these grounds, only small percentages can be used, less than 1% and a somewhat surprising phenomenon has been recently discovered in America that molybdenum forms a compound with the phosphorus eutectic. When phosphorus exceeds 0.12% an eutectic is formed retaining and therefore neutralising molybdenum, and higher percentages of the latter are only partially successful. When the particular service requirements of chilled rolls justify the expense of adding molybdenum or vanadium there is evidence that their use shows a conspicuous improvement in service.

American practice favours lower phosphorus in chilled rolls, but the writer has found that a phosphorus content of about 0.4% is desirable on account of its healing effect by intrusion into the roots of cracks and also into small gas cavities. Many rolls in which a crack has been removed in turning, have been found to be serviceable, although the root, welded by phosphide intrusion, was still visible. In breaking under the tup, rolls with such cracks never broke at the crack.

Copper has been proposed as a cheaper substitute for nickel, but has not been widely adopted. One limiting factor is the moderate solubility of copper in a rich alloy like cast iron, although increased when nickel is present. Experiments have been carried out but the results were indefinite.

One of the factors influencing the use of alloying elements is the need to re-melt worn-out rolls in which the presence of stray elements may be highly undesirable. There is considerable difficulty in using up scrap rolls of varying composition with the object of working up a reasonably accurate analysis of charge for a new roll. Even to dispose of large chilled rolls is an economic problem, since breaking into pieces of suitable size for cupola charging is both dangerous and costly. One effective means of breaking nickel-chromium rolls was found by placing the old roll close to the cupola, so that when the bottom was dropped, at the end of the day's campaign, the hot burning coke buried the roll. In every case the sudden application of heat caused the

roll to break into pieces, which, although ugly to handle, were manageable.

Preparing the Charge

In every case the heavy pieces were melted on the hearth of an oil-fired furnace of 50 cwt. capacity, or in a tilting furnace of 70 cwt. capacity of similar type. Creosote pitch, employed by means of a heated tank and pipeline, was found to be equally satisfactory and not noticeably different from oil, apart from the necessary precautions about maintaining heat and draining pipes at intervals. The charge comprised calculated weights of known scrap together with new iron; approximately three-fifths of the total weight being charged in the hearth furnace and the remainder in the cupola, the latter using lighter scrap and part of the new iron. The proportions for each unit were arranged to suit the

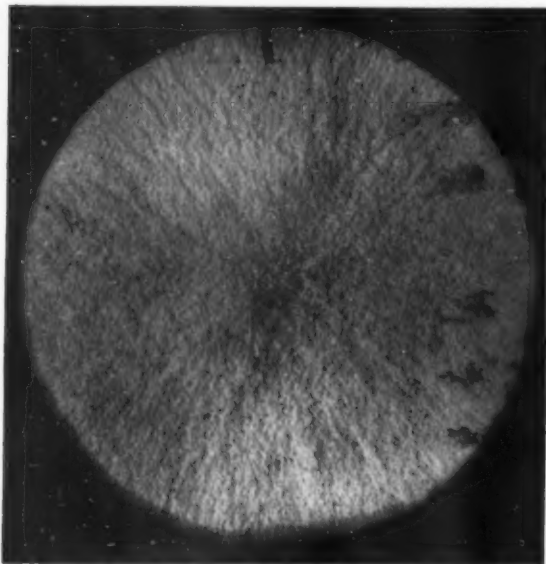


Fig. 1.—Typical fracture of a nickel-chromium chilled roll.

time required to melt the heavy scrap and provide a bath hot enough to receive the cupola metal and to enable a chill test to be made from the mixture.

The nickel to be added to the charge was retained with part of the pig iron for a second charge into the hearth furnace, the nickel being added, in 14-lb. tins, when the metal was definitely melted. No difficulty was experienced in obtaining the maximum yield from the nickel charged in this manner, the tins floating on the surface until the metal was absorbed, when the cupola metal was added. The chromium was added in the form of lumpy high-carbon ferrochrome or briquettes to the cupola charge; parcels were placed in the middle of the earlier charges, an allowance for about 15% loss being made.

After mixing the complete bath and taking a chill test, crushed ferromolybdenum and ferromanganese were added to the stream on tapping into the ladle. This was conveniently effected by means of a scoop fixed on a long handle, the crushed metal being sprinkled into the stream as regularly as possible. Satisfactory yields of molybdenum were also obtained by adding to the cupola stream in a similar manner.

When the whole of the molten metal was accommodated in the ladle and thoroughly stirred, a further chill test piece was cast. Meanwhile the taphole had been made up and, as soon as a chill test had been taken, the metal was returned to the furnace. This step is advisable because alloy rolls require a much higher casting temperature than plain chill rolls, and also to assist complete absorption of the alloying elements, since there is some doubt if cast iron does completely dissolve its constituents. In building up a complex melt from such diverse materials, the results gave a certain amount of metallurgical satisfaction.

Nickel-chromium rolls, it was found, should be cast at a temperature of not less than 1,280° C.; when cast at a lower temperature there was a liability to produce specky surfaces. A curious phenomenon was noticed with rolls of lower nickel and chromium contents, but with manganese content of about 1%. When rolls of this composition were cast below 1,280° C. the top shoulder, when machined, had large flat depressions produced by gas cavities. These obviously would have serious effects on the bearings and so cause rejection of the rolls. This was only noticed with high manganese rolls.

Metallurgical Considerations

Chilled iron rolls are used for so many different purposes in hot-rolling steel and other metals, in cold-rolling steel, non-ferrous metals, rubber, foodstuffs, etc., and receive such diverse treatment in different mills, that only experience can decide which particular type of roll is best suited for each purpose. Broadly, the high alloy rolls are harder and stronger and have all the characteristics of plain chilled rolls, such as sensitivity to sudden heating, to a more acute degree.

With rolls containing over 3% nickel and 1% chromium a chill test piece with a practically white fracture, showing a few grey spots, was found to be most suitable; that is, the test piece was merely a check on the roll being too white. If the grey spots were too numerous there would be too little chill on the roll, according to its diameter. These rolls are regarded as chilled rolls although there is no clearly marked chill, and very little grain in the middle, only an aggregation of numerous grey spots which become fewer and more scattered towards the outer surface. Fig. 1 represents a fracture.

For the purpose of hot-rolling strip steel, numerous casts were made with the higher percentages of nickel and chromium and good results were found in some cases, but early fractures occurred for which no reasonable explanation could be found. For example, one roll might break after one hour in use, and the survivor, cast from the same melt, matched with another roll would give satisfactory life.

The practice of heating all chilled rolls before use is now general, and is even more necessary with the alloyed rolls. For hot rolls it was found desirable to use nickel 3.5%, chromium 1% and in addition to employ some form of heat treatment. One method was to allow the rolls to remain in the mould in the pit until almost cold—i.e., five or six days; this gave some improvement. Another method tried was to place in an almost cold annealing furnace, after rough machining, and raise the temperature over a period of 18 hours to 500° C., soak for 6 hours, and then allow the furnace to cool down over 24 hours. This process greatly reduced the liability to

early fracture but also lowered the hardness somewhat, an undesirable feature after using expensive alloys to raise the hardness. It is true that the abrasion resistance was increased, so that the rolls could be used for longer periods without redressing and consequent loss of production.

On the theory that the austenite-martensite change is incomplete in the rolls as cast, a very long tempering at 300° C. for three days was also tried, with some improvement, but was considered uneconomical on account of the lengthy period of heating, soaking and cooling.

A further method of stripping the rolls hot was much used. At least 2 hours after casting, the rolls were taken from the moulds—an arduous task—and transferred to an annealing furnace heated to 700° C. When the rolls and furnace were uniform, heat was applied and the cooling delayed thereby, taking 36 hours before the rolls could be removed. This gave good results as regards durability, with some loss of hardness.

It would appear to be more satisfactory to strip the rolls the morning after casting, when the temperature is about 300° C. This is much less arduous work for the men, and there is less liability to injury, since the rolls, when stripped shortly after casting, are very tender in the hot condition. In fact, a neck was occasionally pulled off. When stripped the next morning, the rolls are less sensitive to temperature changes and can be charged into a furnace at 300° C. for slow cooling. In fact, there would be more of a hardening and tempering effect by utilising the rapid cooling effect of the chill mould, followed by the tempering effect of the slow cooling in the furnace.

One roll 15½ in. diameter × 32 in. long was required to have a hardness of 80 scleroscope. Efforts were made to keep the carbon contents well up for this purpose, and the composition obtained was C 3.15, Si 0.44, Mn 0.45, P 0.46, Ni 4.04, Cr 1.35, Mo 0.36. After rough machining the roll was carefully heated to 920° C. and then withdrawn from the furnace and hung up by the neck for normalising, the necks being shielded by asbestos-lined sleeves. After cooling, the roll was replaced in the furnace, which also had been allowed to cool. The roll thus treated, after grinding registered 75-76 on the scleroscope instrument. To endeavour to reach the specified hardness the entire treatment was repeated and the hardness raised to 77-78. The roll was accepted, and gave a satisfactory life in a heavy duty mill for cold rolling cupro-nickel.

A subsequent roll of the same size C 2.80, Si 0.50, Mn 0.48, P 0.36, Ni 4.41, Cr 1.30, Mo 0.24 also gave a good life, but it was felt that being so near the verge of possibility, this manufacture entailed too grave responsibilities for regular production.

Among the many rolls cast with higher nickel and chromium contents were small rolls 8 in. diameter for cold rolling. In the cast condition these were too hard to turn with carbide-tipped tools. A part of the barrel was cleaned and gave 97-98 scleroscope. The roll was then annealed at 500° C. and on grinding gave 78-80. Another gave 90-92 as cast and annealed 74-75. It was found that 93 hardness was the limit of machinability. With great difficulty one roll was cleaned on a strip and gave 98-102. The analysis of this roll was C 3.19, Si 0.53, Mn 0.54, Ni 3.93, Cr 1.22, Mo 0.51. The possibilities are indicated that by annealing and machining, and then normalising, a fine hard roll for cold rolling would be produced.

Numerous cold rolls 12½ in. diameter having a composition of C 2.84-3.13, Ni 3.42-4.25, Cr 0.9-1.30 and tempered at 480° C. gave excellent service of two to five years until worn out by frequent re-dressings. Hollow rolls for cold rolling were cast 11½ in. diameter and 32 in. long, but they were found to give inadequate life, fracturing from corrosion fatigue proceeding from the surface of the hollow centre.

A type of roll has been made and known for many years by the term bastard. For the purpose of hot rolling fine steel sections, rolls have been cast in plain iron with only ¾ in. chill. The roll turner cuts off most of the chill so that the section is turned in the very fine zone immediately behind the chill, a fine mottled iron in which the graphite is not noticeable to the naked eye. The application of alloys to this type was found to be very useful. The sections turned easily with a smooth steel-like finish and gave good durability. For this purpose a low carbon content was found to be desirable to ensure very fine graphite in the useful zone.

For convenience in making up a melt, grain rolls were sometimes cast from the same metal with satisfactory results, but since grain rolls frequently give a life of five to seven years the improvement due to the use of alloys was not conspicuous. In fact the plain roll has an important advantage in that the graphite, being in the form of rosettes, gives the roll a dappled surface to the hot metal, which assists in breaking the mill scale and tends to "clean" the steel.

Another proposal to produce harder chilled rolls is embodied in a patent under which lead is injected into the mould after casting. It is known that the chill is formed in less than one minute after casting, and this crust immediately begins to contract, forming a cavity between the roll barrel and the chill mould, filled with gas. The lack of metallic continuity and the insulating effect of the gas are a check upon the rapid cooling value of the heavy mould. The proposal is therefore to maintain metallic contact by filling the space with molten lead. There are difficulties in pumping molten lead on account of its low latent heat and extraordinary penetrating power (in foundry language it is "wick"). By using electrically-heated piping, lead can be pumped in, but in the case of the compound mould used for chilled rolls, means must be found to prevent escape through the sand part of the mould.

To obviate this, pumping was abandoned and a rim of sheet steel, 16 gauge, was introduced under the chilled mould, with the inner edge projecting ¾ in. beyond the mould toward the centre. On casting the roll, the iron bites on to the edge of the steel ring forming a perfect seal at the bottom of the barrel of the roll. On the side of the top box holding the sand mould of the top neck, a small window was cut, and a hopper of sheet steel affixed. In moulding, a small taper bar of steel was placed from the bottom of the hopper to the inside of the sand mould except for a thin layer of sand. After the roll was cast, the piece of steel was pulled out, leaving a passage from the hopper to the neck of the roll. Thirty minutes after casting, molten lead was poured into the hopper until the level indicated that the space was filled. After a time, as the level of the molten lead in the hopper was lowered by contraction, more lead was added, more as a precaution than for any other reason. After two hours, the mould was unclamped and the chill mould lifted, allowing the molten lead to flow away over the steel rim.

On machining, the remains of the steel edge were

easily removed by the turning allowance. Numerous experiments with plain rolls were completely successful. The hardness, compared with normal rolls in the same cast, was raised by 7-9 points—i.e., from 63 scleroscope to 72 without any detrimental effects on service. The process was tried on nickel chromium rolls but found to be unsuitable. With nickel 1.92% and no chromium the hardness was increased by 5 points, 62-67, but rolls with nickel 1.93%, chromium 0.44% and nickel 2.04%, chromium 0.50% were either found cracked and split in the mould on stripping, or they cracked in the lathe on turning, the method was therefore abandoned as inapplicable to alloy rolls.

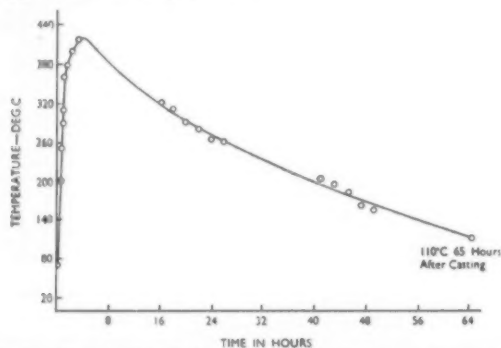


Fig. 2.—Curve showing chill temperatures over a period of 65 hours after casting. Thermo-couple was located under the base of the chill about 1 in. from the chill face.

An interesting metallurgical feature was observed microscopically. The edge of the steel rim embedded in the roll barrel was found to be heavily carburised and raised from dead soft to a high carbon steel, a surprising effect to occur in the 60 seconds of formation of the chill, and short period of cooling subsequently. It was also observed that the molten lead flowed completely away from the sand of the top neck into which it had permeated leaving only slight traces of its presence, a confirmation of the extraordinary penetrating power of molten lead.

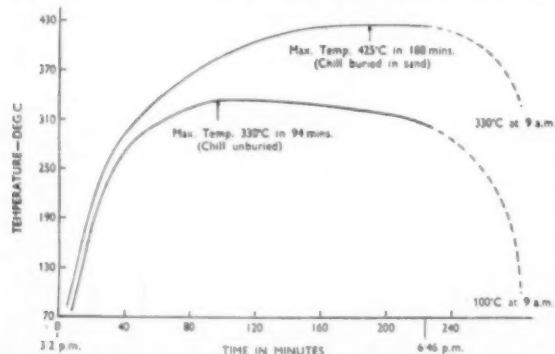


Fig. 3.—Curves showing chill temperatures with the chill buried in sand and unburied, respectively.

By fixing pyrometer couples under the chill mould about 1 in. from the inner face, and another couple fixed to the outside of the chill mould, the whole casting arrangements being buried in sand, it was possible to measure the temperature reached in the heating of the mould. This gives information about the approximate temperature of the roll at a given period after casting,

and provides a guide to the temperature suitable for the annealing furnace receiving the hot roll.

The temperature of the chill mould agrees remarkably well with that recorded by the late Dr. E. Schüz (*Stahl und Eisen*, 1922) obtained under somewhat different conditions. Dr. Schüz actually placed the pyrometer sheaths to be cast in the surface of the roll barrel as well as on the chill mould itself.

The inside surface of the mould reaches 430° C. and the outside surface 330° C. The main value of this information therefore is to correlate the temperatures of the hot roll with those of the annealing furnaces when it is desired to apply heat treatment to alloy rolls immediately after casting.

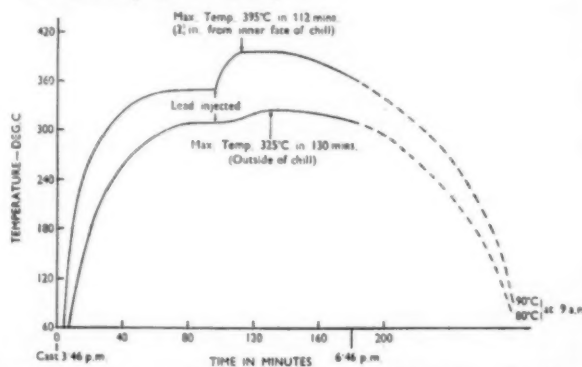


Fig. 4.—Curves showing the effect of lead as a cooling medium on the inner and outer face of the chill, respectively.

Fig. 2 gives the temperature curve for a chill mould in casting, the assembly being buried in sand to produce an annealing effect on the roll. Fig. 3 shows similar curves to illustrate the difference between burying in sand, and the normal assembly not buried. Fig. 4 shows the effect of injecting lead and the maintenance of metallic continuity. With regard to plain rolls the hardness was found to be greater when lead was injected after 30 minutes than after 40 and 50 minutes, indicating a stronger quenching effect. It was found that by burying the assembly in sand, excluding the lead injection, to produce slower cooling seemed to produce rolls less liable to fracture.

Casting

Apart from the necessity of having the metal at a higher temperature, there is little difference between alloy iron rolls and plain chilled rolls. A careful check of the temperature of the metal in the ladle is necessary as there are occasions when it is advisable to return it to the hearth furnace for additional heating. Another important factor is the height of the runner basin. It is obvious that with greater fall the speed of rotation of the metal in the mould is greater. The diameter of the down runner in relation to the size of the roll is also of importance, and generally, the mathematics of the flow of metal in relation to the height and diameter of the runner and to the weight, diameter, and length of the casting, is worth studying.

It was observed that with rolls with unusual length of necks, the casting being about 9 ft. overall, the rotation of the metal in the head persisted for more than one minute, indicating a high speed in the barrel portion. The rolls had excellent surfaces and were entirely free from blemish. The centrifugal action with high-

temperature metal apparently swept away any solid or gaseous matter which might produce cavities in the surface of the roll. It is indicated, therefore, that a higher head of runner than usual is desirable to prevent surface defects.

Since alloy rolls are cast at a higher temperature and possibly with greater speed, the use of a contrivance to release the greater contraction is of importance in rolls other than small ones, and is essential if the length of the roll barrel exceeds twice the diameter. The roll is evidently pegged down at the lower end by the in-runner, and hence the contraction must take place from the top.

The device consists of a sleeve in cast iron with played bottom edge. This edge is machined to fit the chill mould with $\frac{1}{32}$ in. clearance. Cast on the sleeve are four pads to act as bearers for four bolts loosely screwed through the top box. In the sleeve is moulded the top neck, and the chill mould being, say, 4 in. longer than the roll barrel, the arrangement is set so that the top box carrying the sleeve rests on the chill mould, and the sleeve fits inside the chill to give the appropriate length to the roll barrel. This permits the release of the top neck by unscrewing the bolts in the

top box which grip the sleeve, so that the roll can contract freely downwards. For rolls up to 40 cwts. it was found desirable to release the sleeve 20 minutes after casting, lighter rolls 12 minutes, and larger rolls 25 minutes. The clearance between the edge of the sleeve and the chill mould is filled with blacking, although the liquid metal is not likely to escape between two metallic surfaces.

The optical pyrometer was found to be satisfactory for regulating temperature, if any slag was pushed back, and on occasion, when the metal was unduly hot, 1,300° C., there was a waiting period of 5-10 minutes. Generally it was found that by the time the chill test had been taken and the metal tapped into the ladle and skimmed, the temperature was almost correct.

The higher temperature makes more severe demands on the chill mould, and care should be taken to avoid a surge in the flow of metal down the trumpet, because, as in plain chilled rolls, a surge is found to cause an attack on the chill mould with a patch of "sticking." This may cause only surface unsoundness on the roll; on the other hand, it may result in a piece being pulled out of the mould.

To be Concluded

Report of Liverpool Metallurgical Society

By J. A. PARDOE, Honorary Secretary.

THE interest shown in the above Society during the first year has been well maintained during the 1949/50 session and has more than justified its existence as a means of bringing together those whose daily activity involves problems of a metallurgical nature. The membership has been maintained around 100 during its second year.

Five lectures were arranged and the Committee has again been fortunate in obtaining the services of lecturers of high repute in the world of metallurgy, including Mr. G. L. Bailey, Professor C. O. Bannister, Dr. C. H. Desch, Dr. J. C. Hudson and Professor F. C. Thompson, and it is hoped that this standard will be maintained in subsequent sessions.

In addition, joint meetings have been held, at both of which members of the Society were the guests. The first was in November, when by the invitation of the Institute of Welding, members attended a lecture by Professor W. J. Kearton on "The Application of Photo-Elasticity to Measurement of Stresses." The second joint meeting was a sequel to a very successful meeting held in Chester last year, for on January 11th members of the Society were guests of the newly formed North Wales Metallurgical Society at a lecture given in Shotton by M. W. Thring, Esq., M.A., on "Recent Developments in the Study of the Flow of Steel." This lecture was preceded by a visit to the Hawarden Bridge Steel-works by the kind invitation of the management of Messrs. John Summers, Ltd.

Another works visit, that to British Insulated Calender Cables, Ltd., Prescott, on April 27th marked the close of the session.

Recently an offer of affiliation was received from the Council of the Iron and Steel Institute, and this was accepted by the Committee of the Society. It was mutually agreed between the Institute and the Society that the Institute would:—

1. Encourage its ordinary members or associate members

in the area to become members of the Liverpool Metallurgical Society.

2. Accept members of the Liverpool Metallurgical Society as associate members of the Iron and Steel Institute at the reduced annual subscription of £1 11s.

3. Welcome members of the Liverpool Metallurgical Society at its technical meetings.

4. Make its papers available to the Liverpool Metallurgical Society for discussion at local meetings.

Whilst for its part, the Liverpool Metallurgical Society would:—

1. Encourage its members to become ordinary members or associate members of the Iron and Steel Institute.

2. Invite members of the Iron and Steel Institute resident in this area to attend meetings of the Liverpool Metallurgical Society.

A new side to the Society's activities was foreseen in the proposal made by several members that some form of social evening should be held to conclude the session. A questionnaire has been sent out in February and it is hoped that sufficient replies will be forthcoming to make the venture a success.

In concluding this brief report of the 1949/50 session may I express the hope that 1950/51 will see a further strengthening of metallurgical interests on Merseyside. It is hoped, for example, that as many members as possible will avail themselves of the advantages of the Affiliation with the Iron and Steel Institute; interested readers may obtain further information on request to Mr. J. A. Pardoe, 183, Queen's Drive, Liverpool, 4.

Acknowledgment

The Society records its thanks to the Liverpool Engineering Society for again loaning their room for lectures and to all members who by their presence and by discussions ensured the success of the meetings. Appreciation is also recorded of the work done by Mr. Gellatley, who retired from the Secretaryship in December after being in a large measure responsible for the success of the inaugural session; and to all officers, in particular, the President, thanks are due for their help and advice.

That Specification : Master or Servant ?

With Particular Examples from the Cold-Rolled Non-Ferrous Industry

By Thomas B. Crow,* Ph.D., M.Sc., F.R.I.C., F.I.M.

While the need for standards was never greater than to-day, slavish adherence to them frequently leads to trouble due to a lack of knowledge of the working characteristics as applied to the work in hand. Nowhere is this emphasised more than in the case of the cold-rolled non-ferrous materials, and although the numerous examples cited by the author are taken from this field, much of the argument in this provocative article is applicable in a wider sense. Opinions must necessarily differ on such a contentious subject, but there can be no doubt that with fuller knowledge both of the particular purpose for which the material is to be used and of the fabricating technique to be employed, material could be supplied to a standard more satisfactory to all concerned.

ALTHOUGH this article has been written primarily with a view to helping those who may be concerned with the manufacture, supply, testing and applications of certain cold-rolled non-ferrous metals, it may be not without interest to the general reader, for, as its principal title indicates, it deals with a problem which exists in many branches of industrial metallurgy. The temptation to write it originated from the author's experience that a good deal of dissatisfaction might arise when attempts were made to apply the "Specification Habit" (defined below) to certain cold-rolled metals and to the components manufactured therefrom. The causes for this are so widespread, and may arise from so many quarters, that it is not possible, in a short account, to survey or even tabulate them with any degree of precision or completeness. There would be little exaggeration, however, if it were claimed that in no other branch of metallurgical industry is there so much scope for mis-understanding, mis-interpretation and abuse of "The Specification." The reason for this will become clear as the reader proceeds; it is due to a lack of appreciation of a fundamentally important fact—namely, that the subsequent fabrication operations to which the rolled metal will be subjected, and the ultimate uses to which it will be put, are so varied, and often so severe and intricate, that in order to achieve both successfully, such unpredictable and unsuspected combinations of properties are called for that no ordinary general specification could possibly encompass them. That statement does not mean that existing specifications are of no value and should be scrapped forthwith. It does, however, mean that they do not automatically furnish their adherents with the same guarantee of suitability and safety as they would expect, and get, from so universally accepted a specification as that for, say, a mild steel boiler plate, for use as such.

Objects

More especially, the objects of this article are to indicate :—

1. The pitfalls of unenlightened reliance on specifications.
2. The limit of their usefulness.
3. The too-frequent dis-service of inflexible inspection procedure.
4. The need for revision of certain B.S.I. specifications, as such,

and, generally, to suggest means by which these shortcomings may be overcome to the greater satisfaction of all concerned. The treatment of the subject is illustrative and, in certain respects, piecemeal; no claim is made that it is exhaustive; neither should the suggestions for revision of specifications be regarded as comprehensive. The controversial nature of this article has necessarily led to what appear to be inconsistencies, since opposing view-points have had to be presented. The final resolution of these, however, has been attempted in the writer's concluding remarks.

Definitions

For the purposes of this article, the following shall apply :—

THE SPECIFICATION HABIT is an attitude of mind towards specifications. It is more easily experienced (and sometimes accurately appraised) than defined; in fact, a large part of this article is occupied with a definition *in extenso*. For immediate purposes, however, the following will serve: it (the S.H.) is the practice of regarding the association of a named specification with a particular job as an insurance (the specification being the "policy") whereby the insured is indemnified against the supply of unsuitable material. The craving for this insurance—this shifting of personal responsibility on to the specification itself, whereby it becomes a convenient scapegoat for someone's lack of due consideration for the essentials of the job—is frequently accompanied by carelessness in the choice of the "policy." But, having made the choice, rightly or wrongly, the question of suitability fades right out of the picture. For, once the specification is chosen, it somehow or other (by the most extraordinary operation of wishful thinking) becomes divinely invested, as it were, with the unquestioned virtue of suitability and must therefore be accordingly revered and complied with, down to the very last letter of its provisions, be they never so inapplicable!

THE PURCHASER is the organisation in whose factory the rolled metal is cold formed, into pressings, piercings, spinings, stampings, springs and such like; the terms "press shop" and "stamped brass-foundry" are used in a wide sense; the latter term includes items made also in copper and/or in phosphor-bronze. The purchaser may buy direct from the ROLLED METAL MANUFACTURER or from a MERCHANT. The products of the purchaser

* Emery Bros. Ltd

pass to the CONSUMER, who may be "part of" the purchaser, in another department of the same factory, or who may buy from the purchaser. THE INSPECTOR may act for the manufacturer, purchaser or consumer, or for the ULTIMATE USER, who may be a builder of battleships, or a child with a mouth-organ. Inspectors and their staffs are often possessed of the specification habit, sometimes with unfortunate or annoying results.

SHEET, STRIP AND FOIL are particular categories of COLD-ROLLED METAL, though the author has used the word "strip" rather widely in the earlier part of this article. COLD-ROLLED FINISH should be distinguished from ANNEALED FINISH.

PART I

The Relation of the Specification to the Job

1. Broadly speaking, there are two rather diametrically opposed views or definitions of the word "Specification." In a nutshell, these may be very aptly expressed by the words "Master" and "Servant." Most specifications are statements of chemical, physical and mechanical (and maybe other) properties which the strip is required to possess, to stated values. The simplest of all these may be exemplified by quoting from a very typical form of purchase order—namely, *Half-hard Brass*. At the other extreme there is the voluminous document, which is usually designed by purchasing organisations originally for their own internal technical use, but which has become, by long usage, a device mainly to safeguard their metal purchases. Intermediate between these there are the British Standard Specifications—intermediate, that is, in length, reasonableness and precision.

2. There is usually one characteristic feature shared by all of the specification types already mentioned: namely, the absence of any reference to the kind, or to the function, of the component to be made from the strip.¹ This omission is very largely responsible for the *insidious* growth of an attitude towards specifications which ultimately leads to their becoming the *master* (see def. "Specification Habit"); this is particularly so when the application of the specification is in the hands of academically minded personnel, who are most seriously conscientious in regard to the strict observance of the letter of the law.

3. Now let us consider the other view-point—that in which the specification is the *servant*. In the days before the existence of the specification, the only criterion of satisfaction in purchasing a material was, "Will it do the job?" Gradually, it was established that a material which *would* do the job could be expected to conform to certain numerical values for chemical composition, tensile strength, Izod number and so on. If these values were collected together into a "statement," they could be accepted with confidence by the purchaser as a guide or yard-stick when buying his material. As such the statement was his *servant*. Since those days a change of attitude has grown up, as already mentioned, a change which is much for the worse, in some respects, in many sections of metallurgical industry and particularly so in the one under present notice. (Although the author's experience has been mainly limited to brass, copper and phosphor-bronze strip, the observations made and the principles advocated in this article are of much wider application, both inside and outside of the cold-rolled industry).

4. It has been stated that in the early days of specifications there was a close connection, which really meant something, between "specification" and "job"; the two have now become virtually separated. There are, to-day, several specifications for cold-rolled strip which, *considered as "matters apart,"* are (notwithstanding certain shortcomings to be dealt with later) excellent, but they have been drawn up in such a way as to be only partially successful in offering the maximum guidance to the purchaser. They savour somewhat of a collection of water-tight compartments (i.e., tempers) *into one of which the job has to be fitted*. Thus, the rolled metal manufacturer has to make his metal to suit a specification rather than to suit a purchaser's job, and so it (the specification) becomes the master. Although it is true that in a great many cases hundreds of varieties of pressings can be made satisfactorily from strip which *does* strictly comply with the provisions of one of these water-tight compartments, it sometimes needs but a chance combination of circumstances in a purchaser's work (for example, the engagement of a new press-shop executive), to reveal that *that* particular water-tight compartment was not the *best* one, although its choice had hitherto given what was regarded as complete satisfaction. Let this point be illustrated by an example. Suppose a purchaser, being a maker of some form of electrical control gear, wishes to make, in his own press shop and for his own consumption, say 1,000 small brass pressings. He places an order with a cold-rolling mill for so many cwt. of brass strip, maybe to B.S. 265, quarter-hard temper. In due course the metal is delivered and is received into the "goods inwards" inspection department. It is there subject to the prescribed tests for B.S. 265, quarter-hard, and found to be definitely in the half-hard temper. Now, one of two things can happen. Either the metal will be rejected out of hand and returned to the manufacturer "without the option," or, if production in other departments is being seriously held up for want of the thousand stampings, somebody (usually the metal roller) will suggest that, notwithstanding the report of the goods-inwards inspector, the metal be given an actual fabrication trial in the press-shop. This suggestion is reluctantly acted upon and, to the complete surprise of everyone (except the manufacturer), the half-hard stuff is found to make a better pressing than the quarter-hard, whose suitability had never even been in question.

5. And so, notwithstanding non-compliance with the specification, a superior and thoroughly satisfactory stamping is made; "Exports" get their control gear on time and everyone is happy. But the "goods inwards" inspector can best serve his company by taking immediate steps to see that the buying department does not order any more quarter-hard brass for that job. He can also see to it that the remaining stocks are preferably diverted to more suitable uses. In passing, it may be mentioned that some purchasers keep a few "stock" tempers, *and those tempers only*, in their stores, and make them fit all their jobs. Such practice is reminiscent of the doctor who kept six stock medicines only and made all his patients' illnesses fit them!

6. Another instance of an ill-suited specification may now be cited: an instance in which the strip will not make the pressing. Let the trouble be traced to its source—namely, the designing department of a purchaser firm making (say) an article which contains a phosphor-bronze spring electrical contact of complicated shape,

¹ "Copper for General Purposes" is the title of a specification which suggests its possible *unsuitability* for some particular purpose, for which it may have been unwittingly but optimistically chosen.

involving some severe forming (bending) operations in the presses. The details of design are worked out by a draughtsman. He has in his mind "Spring phosphor-bronze." He consults "the book of Specs.," and finds "Phosphor-bronze, B.S. 407, Grade III, Extra-hard temper" which he then and there, without consulting anybody, decides is the right metal for the job. Accordingly he enters these details on his drawing, and also states the width and gauge. In due course, the metal arrives; it passes through "goods inwards" as O.K. and is passed to the press shop where it meets with surprisingly disastrous results—the "blanks" won't "take the bends" but crack and split up in a most disconcerting fashion. "Goods inwards" re-tests the metal and confirms that it is "perfectly all right"; that is, it is "what was ordered." . . . It hardly seems necessary to point out that the difficulty here had its origin in a drawing office, which was not too familiar with the properties of rolled phosphor-bronze. So far as design of pressings is concerned, some drawing offices seem to be lacking in appreciation of the following facts concerning the temper² of cold-rolled metal supplied—namely:—

- (a) High values for springiness, tensile strength and hardness are opposed to formability; in most forming jobs, a compromise must be sought. (See (b) below).
 - (b) Rolled metal has appreciable directional properties. The direction of the grain in a pressing is of the greatest importance in determining where the compromise (see (a) above) will occur. By a suitable set-up of the press tools in relation to the direction of the grain, considerable increases in formability can be attained without appreciable loss of springiness and strength. (See (f) below).
 - (c) Increasing the radius of curvature of bends lessens the tendency to cracking on those bends and so permits the use of a stiffer and stronger strip.
 - (d) Press operations themselves frequently cause appreciable work hardening of the metal. It must be distinctly understood, therefore, that where a specific temper is called for on a drawing, as applying to metal as ordered, it shall not also apply to any pressings made therefrom.
 - (e) Temper is mainly conferred upon a metal by mechanical working, and not, to any extent, by heat treatment. There is an erroneous belief that temper is a matter of heat treatment (as with steels) and that metal which is off-temper can be corrected by re-tempering. Metal which is *slightly* too soft, can be hardened by further rolling, but only at the expense of a slight reduction in gauge.
 - (f) Where a consumer firm orders its requirements in pressings from more than one outside supplier, it should be borne in mind that differences in press shop technique sometimes call for rolled material of different temper qualities to make the same article. In any case, it should be left to the makers of the pressings to do their own specifying and ordering.
7. A third instance of the limitations of specifications may now be given. A consumer firm, making hundreds of thousands, or perhaps millions of one kind of pressing only, orders its requirements of material to a specifica-

tion, from four different rolling mills. The press shop foreman reports that metal from three suppliers is O.K., but that that from the fourth "just won't go nohow," notwithstanding that it is *identical* with the other three, as seen through the eyes of "goods inwards." The explanation in this case is quite simple; it is that the upper and lower values for composition and properties, as given in the specification, are so wide apart and permit of so much latitude, that a particular combination of these values (e.g., Vickers Hardness in conjunction with tensile strength and phosphorus content—if a bronze) is sufficient, or more than sufficient, to cause failure in the presses. In other words, the specification is too crude, too arbitrary and too incomplete to reveal just those fine shades of difference in properties which, taken as a whole, are so telling in the press. Obviously the only satisfactory test of suitability is an actual press trial.

8. There is another aspect of ill-suited specifications. Press shop operations and conditions (tool clearances, in-between anneals, lubricants, and other factors) vary considerably from purchaser to purchaser, even when making the very same component. Hence, in the matter of the properties of metal supplied to each one, what is one man's meat is another's poison. Individual purchasers' needs must therefore be studied.

9. The four instances which have just been described afford examples of dissatisfaction which has primarily arisen through lack of knowledge, co-operation or sufficient attention to the individual requirements of the job. In the first, metal of "second-best" temper had been repeatedly ordered, by custom of the buying department. In the second, the drawing office "did not know." In the third and fourth, it was taken for granted that the choice of a specification automatically conferred a guarantee of suitability. Many more instances might be given, all of which, including the four above, fall into one category—namely, misapplication or mis-interpretation of specifications as they are. Considering specifications as instruments intended as a guide for the press shop superintendent, they contain a good deal, which, unless accompanied by a lively appreciation (by him) of the potentialities and limitations of strip for cold working, can only lead to frustration.

PART II

Ambiguity of Temper Designations

10. It might be inferred from the last paragraph that the specifications themselves were perfect and that the shortcomings always lay entirely with the people who used them. If this were so, the present author could wind up this article here and now with a suggested procedure, as between rolled metal manufacturers and purchasers, for making the existing specification a useful servant to the latter. If all specifications, considered simply as catalogues of chemical and mechanical properties of cold-rolled strip, were perfect, then the dissatisfaction, which is the theme of this article, would at least be halved.

11. It therefore becomes necessary to make a detailed examination of some of these specifications. The simplest of them consists of a statement of temper; the purchaser asks for so much metal—be it brass, phosphor-bronze or copper—in a temper which he designates as dead soft, soft, quarter-hard, half-hard and so on up to extra spring-hard. What do these terms signify?

² And other properties, to which reference will later be made.

The purchaser knows (more or less) what *he* means by half-hard, but does the manufacturer know what this purchaser means? Tradition says that the customer (purchaser) is always right. Hence the manufacturer must, in dealing with *that* purchaser, adopt his (the purchaser's) nomenclature, regardless of his own views in the matter. The British Standards Institution has laid down (within certain limits) a *fairly* precise interpretation of some of the above terms, but this again may be different from that of manufacturer *and* of the purchaser and in any case there is a very large number of purchasers who fight shy of "B.S.I."

American Practice

12. The American specification for "Sheet High Brass" gets over this ambiguity very easily. The Brown and Sharpe range of gauges, from 0.005 in. to 0.460 in. is based upon a geometrical progression.³ Any gauge (expressed in inches) in the series will be found by multiplying the immediately preceding gauge by approximately 1.123. This ratio is called "One Gauge Number." Tempers are expressed as so many "gauge numbers hard." Thus, quarter-hard temper is always rolled "one gauge hard"; hard temper is always "four gauges hard" and so on. Table I briefly illustrates this.

TABLE I

Finished Gauge	Gauge* from which the (soft) metal must be rolled to give		
	Qr. Hd. (1 G.N.H.)	Hard (4 G.N.H.)	Extra-Hard (6 G.N.H.)
0.0201	(10.9%) 0.0201 × 1.123 = 0.0225	(37%) 0.0201 × (1.123) ⁴ = 0.0320	(50%) 0.0201 × (1.123) ⁶ = 0.0402

* In inches. G.N.H. = gauge numbers hard.

The advantages of such a system need no emphasis; a *fixed* percentage reduction is applied for all gauges in any particular temper.

13. The author has encountered instances of the use of the terms "soft" and "dead-soft," to denote degrees of liability to "orange peel," whereas, of course, the more usual significance of these terms is "drawability." And so one might go on. The purchaser assumes that the manufacturer uses the same language as he does; he is surprised, indeed, to learn that there *is* any other language. The result of it all is vagueness, uncertainty and, may be, a delivery of unsuitable metal. The chances of this may be greater if business passes through the intermediary of a merchant or stockist; for he, again, may have *his* own language or may even be inarticulate!

Specially Compiled Specifications

14. Some of the larger purchasers or consumers of strip have produced their own specifications, copies of which sometimes accompany their orders. Such documents are the result of attempts (1) to present a set of requirements which *they think* will suit their own particular range of jobs (or even to suit one job only, repeated in hundreds of thousands) and (2) to protect themselves (for there is the usual "goods inwards" inspection procedure) against deliveries of material which they claim "does not comply." These specifica-

tions appear in some cases to have been drawn up by technicians who are not over-conversant with what can be reasonably expected of the average rolling mill. They are usually modelled on B.S.I. lines, but some of the maximum and minimum values of those specifications, whether of dimensional tolerances or of physical properties, may be altered, or additional clauses may be inserted, so that the specification becomes practically useless. Some specifications contain *metallurgical* impossibilities; others contain *commercial* impracticabilities. Again, in others, some of the provisions are even unnecessary for the purpose for which the specification is drawn up. Technicians who are charged with the drawing up of special specifications should note that those of the B.S.I. have been prepared by practical metal rollers of long experience and that any appreciable overstepping of *their* provisions in the matter of tolerances, or by the insertion of freak or over-stringent requirements, should only be resorted to on direct evidence of real necessity, and even then only with manufacturer's assent.

15. Some of these purchaser's specifications suffer from omissions—something that *does* matter is left out. A striking instance of this sort (within the author's own experience) resulted from a purchaser's omission to tell the manufacturer beforehand that a pack of 50 thicknesses of 0.001 in. thick copper was required to fit into an inspector's (goods inwards again) "go" gauge of 0.050 in. opening. It is just not fair for manufacturers to be kept in ignorance in this way, and then to be upbraided afterwards.

In the concluding part of this article, which will be published in the next issue, extended comment will be made on some B.S.I. specifications and the author's conclusions will explain the two lines of thought which are apparent in the discussion

H. F. Furnace for the Norwegian Government

Among the high frequency heating equipment which the General Electric Co., Ltd., has recently exported to Norway is a 5 k.w. induction furnace specially designed in the Company's Research Laboratories for metallurgical research by the Norwegian Government. The furnace is housed in a sheet metal cabinet with a top of heat-resisting material. A lever operating through a notched gate is used to raise or lower the crucible and during operation the furnace is covered with a lid which has a small hole to allow observation of the charge or the insertion of a pyrometer. The lid, table top and removable rear panel are all fitted with safety switches to prevent power being switched on when they are not in position. Two crucible assemblies are used with the equipment, a graphite crucible for melting metals with which contact with graphite is permissible, also for melting non-metallic charges: and a refractory crucible for ferrous charges.

Power for the furnace is supplied from a standard G.E.C. 5 kw. high frequency heating generator and typical performance figures for the plant are:—copper, 4½ lb. (2 kg.) raised to melting point in about 15 mins.; steel, 2½ lb. (1 kg.) melted in 12–15 mins. The overall power consumption of the equipment when in full operation is 10 kw. from a 400–440 volt, three-phase supply.

³ The B. and S. series of gauges starts at 0.005 in. and finishes at 0.460 in. It contains 40 sizes. Treated as a geometrical progression, we have—
0.460 = 0.005 × 1.123³⁹
whence $r = 1.123$ approx.

Higher Technological Education

By G. Tolley, M.Sc.

In the United States the development of technological education has proceeded hand in hand with the growth and development of industry. The general educational plan in this country differs in many respects, but in pointing out the responsibilities of the Universities and industry, particularly the former, in raising the standard of technological education in Britain, the author draws on his observations of the American system.

THERE has been considerable controversy recently amongst leaders in University education as to the means to be adopted for increasing the general standard of technological education in Britain. Virtually everyone connected with higher education is agreed that such a development is long overdue, but there is singularly little agreement as to the exact process whereby this shall be accomplished. Fingers of admiration have been pointed at the twin pinnacles of technological education in the United States—the Massachusetts Institute of Technology and the California Institute of Technology—and much has been said as to the applicability of the American model to Britain. There have been representative voices from practically every quarter concerned with this problem—from the Universities, the Government (the Percy Committee), the technical colleges and the House of Lords—and many important personages have had their say, and will, no doubt, have it again and again before any final scheme of development is decided upon. There is one section of responsible opinion which has, however, not yet expressed any views on the matter, and which has not been asked to do so. In fact, this particularly vital section of the community, which is concerned with technological education far more than any other, has been almost completely ignored, as though it had nothing to do with the question. I refer, of course, to industry, which will, in the main, absorb technologists and technological advances, and which will ultimately decide whether it is worth while educating for technology or not. It is because industry is the most vitally concerned party in the business, that I wish to make a few observations relative to the development of American technological education, which has proceeded hand in hand with the growth and development of industry; I also wish to comment, as an industrial research worker, upon the need for a close scrutiny of present-day higher education in Britain.

Technological Education in the U.S.A.

It has been my privilege for some little time to have the opportunity of working in the U.S.A., and of observing educational practices, and the relation between industry and educational institutions. There is no need to stress the high standard of technological education in the United States; this has been, and is continually being, done by numerous admirers. Institutions such as M.I.T. and the California and Carnegie Institutes of Technology are, in every way, models of the way in which technological education can be fostered. These institutions are undoubtedly the greatest of their type, and yet their sterling qualities are not, perhaps, as fully realised as they might be. Their names may tend

to mislead, and one of the difficulties in facing up to the present problem in Britain is concerned with the fact that they are nothing more or less than Universities of the highest calibre. If this fact is lost sight of, as it very often is, then argument tends to accomplish nothing. Again, it should indeed be asked why this development has proceeded to such an extent in the U.S.A., and hardly at all in Britain. The question is a complex one, but one answer is that the bulk of scientific education in the U.S. leans heavily towards the technological side, whereas in Britain this is not so. For one institution such as M.I.T., there are, in the United States, some dozens of poor relations in the form of Universities and colleges which have become bogged down in the non-productive minutiae of technological detail, and whose educational standards are low because they have not achieved such freedom of thought and independence of action as the three oft-quoted institutions already mentioned. When considering the highest that America has to offer, it should not be forgotten that there are failures—failures which arise from the fact that, on the one hand, the institutions concerned have not learned the secret of technological education, whilst on the other, they cannot break away from technology and go over to the paths the British Universities so conspicuously tread. In other words, the dangers of technological education are also plain to be seen in the United States, and they should be considered along with the successful attributes associated with the prestige of the established Institutes of Technology.

These successful attributes are items which must be translated into any British scheme, and it is well to mention a few of them so that we may see what is required; then we may consider how it may be achieved. In the first place, top-ranking technological institutions in the U.S.A. have managed to preserve an independence of outlook and a forthrightness of spirit which are the envy of some Universities. They are, in every sense, capable of giving to their student body a thorough grasp of principle and a freedom of approach which ensures the continuation of a scientific attitude throughout the career of the technologist. Their relations with industry have been close, but not stifling; each has helped the other in an admirable fashion, and quite apart from financial support from industry, there has been a moral support which is significant because it helps to demonstrate the true partnership which must exist if there is to be successful education in the realm of technology. Post-graduate work has always been given a pride of place, so ensuring the continuation of productive effort in these institutions, and attracting able and constructive minds to maintain this tradition. Buildings and equipment are excellent, but that it not to say that

there is no lack of room, or no makeshift methods. Indeed, one can still find a "string and sealing wax" outlook in an American Institute of Technology, much as one finds it in a British University; if this were not so then research would be stifled and projects would be interminable.

The American model is founded, therefore, upon the idea that technological education can only be successful if based upon the foundation of scientific principle, which will be sufficient to ensure that the student is fully aware of his responsibility in applying theory to practice, and vice-versa. Stated in these terms, it is difficult to differentiate the Institute of Technology from the American University, for the educational aims of the latter are truly pragmatic, and, indeed, it is because of this that the former has developed to such a large extent. It should not be forgotten, therefore, that the educational background in the U.S. is somewhat different from that in Britain. For this reason, a mere transplanting of an M.I.T. to British soil would be likely to bring more problems in its wake than it actually solved.

The Role of the University in Britain

In Britain, due possibly to a reluctance to shake off the shackles of Victorianism, the Universities still hold a privileged position of academic freedom and independence of action—and justly so—whilst technology has somehow continued to be identified with the training of technicians—of tradesmen. The latter, erroneous concept, and its firm establishment in the minds of many educationists, is certainly one of the reasons for the present controversy, in which University professors, and leaders in University thought generally, are trying at times to walk a precarious tightrope between the principles of University education and the aims of the New Technology. In fact, it is precisely because University education of to-day has lost many of its principles, and is so unsure of its own aims, that our University leaders are incapable of giving a lead in the development of technological education. Neither do they see the responsibilities which they have in fostering the growth, not only of technology, but also of a firm interest on the part of industry. Technological education cannot grow, cannot produce good material, unless there is an intelligent attitude in industry, and unless there is a really progressive outlook on the part of industrialists who are going to demand satisfaction from technological institutions. As the cost of labour increases in Britain, there may be a greater desire in industry to apply technological advances as they should be applied, and in this way there would be a tangible, yet not restricting, partnership between industry and higher technological education, just as there now is in the U.S.A. It would seem also that the Research Associations in Britain, which have developed along lines virtually unknown in the United States, have an important part to play. Their liaison might take the form of seconding personnel and ensuring adequate joint discussion at every opportunity, thus providing a firm basis for mutual growth and development. There could be help here, too, from the Government scientific departments, and also from the Universities.

First of all, then, it behoves Universities to formulate clearly what they regard as the distinctive characteristics of a University education. The spiritual and moral values implicit in such an education, will, once more, have to be restored to their rightful place, and there

will have to be less talk about a mere academic freedom, which is, so often, meaningless. Secondly, Universities will have to adopt an attitude which is helpful to the growth of higher technological education in Britain. If Universities have one task of importance, then that is surely concerned with leadership in the whole field of higher education. That is not to say that they must be directly responsible for technological education, but it does mean that this question must be discussed on the basis of deciding much more than whether separate institutions are to be set up, specifically adapted for this purpose. It is sufficient to say that the present position of many of our technical colleges, some of them with fine potentialities, is not what it should be because Universities have been all too eager to ignore poor relations, and turn the other way when these colleges have been held down by crippling restrictions of local authorities. By a firmer co-operation between the two, technical colleges could yet play a much greater part in higher technological education, as is apparent from the example of the Royal Technical College.

The Industrial Aspect

On the other hand, the attitude of industry will, to a large extent, decide the success of any scheme, for it is industry which must be served, and unless it is a good master little will be accomplished. It is desirable that the Federation of British Industries should extend its admirable original survey of research in technical colleges, and consider just how these institutions may be expanded or modified to meet the needs of industry. In this way, by considering practical demand, it may be possible to fit these into an integrated picture, in a manner more satisfying than that of the Percy Committee. There is no doubt that, given the blessing of the Universities, several technical colleges could be aided to the point where they would be capable of assuming responsibility similar to that of the Universities, but in the field of technology. There is an example in the case of the Central Technical College in Birmingham, in close proximity to a University of acknowledged stature; the situation is still more promising when one considers that new buildings are now under construction for the former, and that its geographical position is such as to make it an ideal centre for higher technological education. Here is an opportunity for experiment which could yield valuable results. If Universities feel, as many of them do, that their primary responsibility lies outside the field of technology, then it will certainly be necessary to develop other institutions, and this will, probably be the only practicable solution. However the contribution which they can make to the growth of higher education on technology is paramount, provided that they first put their own house in order, and assume the moral leadership which is their birthright.

Industry also can provide an answer to many of the questions now being asked, by considering in earnest, for the first time, its own responsibility to education, and by pointing out some of the qualities desirable in technologists of the first rank, who are so badly needed. As long as Universities are turning out specialists who are neither first-rate technologists, nor scientists capable of thinking clearly for themselves, there will be a crying need in industry, and, indeed, in the whole community. In looking at the United States the Universities have perhaps allowed their perspective to be falsified by considering only high calibre institutions, by not taking

into account sufficiently the differing patterns of industry in the two countries, and further, by allowing themselves to be deluded into thinking that the highest qualities of a University education are really manifest in the methods and products of to-day. The failure to consider in all its aspects the relation between higher technological education and industry in the present-day conditions in Britain is an indication that the leadership which should logically be given by the Universities is not at the moment apparent. It is necessary, therefore, that

there should be not only a close study of the American method—taking into account industrial relationships—but also a close examination of British needs, together with the methods at present available for satisfying these needs. It is a pity to see industrialists and educationalists alike repeatedly sending messengers to the oracle of the New World, when there is surely sufficient strength, initiative and independence left in the Old to think and work out a scheme which can give satisfaction to all concerned.

Costing and Modern Accounting Methods in the Metal Industries

VII.—Ascertaining the Balance of Taxable Profit

By S. Howard Withey, F.Comm.A.

Probably the most complex part of accounting in modern industry is that of ascertaining the balance of taxable profit, which is discussed in this section. Considerable adjustment to the profit and loss account may be necessary before the Inland Revenue Authorities accept the net profit figure; many items of expenditure are not allowed while others must be claimed as will be noted in the author's consideration of the factors involved.

AFTER all management and working expenses have been charged against the revenue, including the cost of maintaining the plant, machinery and other productive assets in a proper state of repair and efficiency, also an adequate sum to cover the decline in the capital value of the profit-earning assets employed in the business, the amount by which the total of the items shown on the credit side of the periodical profit and loss account exceeds the debit total should represent the balance of net profit which has actually been realised; in the event of the total of the items shown on the debit side of the account exceeding the credit total, the difference would represent a financial loss or deficit.

In the case of the metal manufacturer or trader in business on his own account, an amount equal to the profit balance may be withdrawn from the business as and when required, or, of course, it may be retained in the business and added to the amount of the proprietor's capital for the purpose of strengthening the financial structure and provide the funds needed for expansion or modernisation. If the business is being operated by two or more partners, the balance of net profit shown in the final audited account would usually have to be shared by the partners in strict accordance with the terms and provisions of the deeds or other working agreements entered into, the amounts due to each partner being usually merged with their capital accounts; but in the case of the private or public limited liability company the capital must be shown quite distinct from profits, as it cannot be altered unless a number of definite statutory requirements are fulfilled. Moreover, only the figures required to determine the amount of profit realised, or the extent of the loss which has been sustained, during the particular period under review, should appear in a company's profit and loss account, the balance, if any, which has been brought in from the previous year or period, also all appropriations and allocations being enumerated in a special account. For example, the directors of an engineering company in the North recently submitted an appropriation account made up as shown in the accompanying table.

The net profit figure shown in an ordinary profit and

loss account may have to be subjected to very considerable adjustment before being accepted by the Inland Revenue authorities for the purpose of computing the balance of taxable profit under Schedule D of the income tax code. Some of the items which have been debited in the ordinary way, although representing definite charges against the periodical trading revenue, will not be permitted to stand as deductions for tax purposes, while, on the other hand, under certain conditions allowances may be made in respect of expenditure which does not appear in the final profit and loss statement.

Allowances

Amounts which have been written off the book values of large and small furnaces, forging presses, rolling mills, and other units or sections of equipment, including accessories, auxiliaries and fitments, etc., to cover decline of capital value arising from wear and tear, corrosion, obsolescence, effluxion of time or other deteriorating factors, are not necessarily legitimate deductions from the taxation standpoint, as the allowances actually granted are arrived at by reference to a definite scale applicable to the particular industry or trade. These scales of wear and tear allowances are subject to alteration from time to time in order to satisfy the needs of taxpayers and industry in general, and are intended to cover those parts of the machinery and plant which have actually been worn out in the

PROFIT AND LOSS APPROPRIATION ACCOUNT
Year ended March 31st, 1950.

Debit.		Credit.	
To Dividend on First Preference Shares, less income tax...	£ 8,750	By Balance brought forward from 1949-50	£ 28,719
" Dividend on Second Preference Shares, less income tax	10,000	" Net Profit for Year, transferred from P. and L. Account ..	63,815
" Interim and Final Dividends on Ordinary Shares, less tax	8,000		
" Provision for Deferred Repairs	10,000		
" Staff Bonus	5,000		
" Reserved for Contingencies	7,500		
" Transfer to General Reserve	12,500		
" Balance carried forward to 1950-51	30,784		
	£92,534		£92,534

process of earning profits to be taxed. Moreover, the allowances are not granted automatically but must be claimed, and among the items of expenditure which are not allowed are the following :—

The capital cost of acquiring any site, or the rights in or over a particular site; the cost of acquiring mineral deposits or rights in or over them; the cost of any assets which are treated as such for taxation purposes; the cost of constructing works for the processing of the products of a deposit, as distinct from the preparation of raw material for use as such; the cost of erecting buildings for use as offices, including a proportion of the cost of other buildings which are used as offices where the cost exceeds one-tenth of the total expenditure involved in the erection of the entire structure.

Initial allowances may be claimed in respect of sums expended in the construction of works which are likely to be of little or no value when the source of deposits is no longer worked, and so long as the assets have not been sold in the meantime the allowances are also available as regards expenditure incurred between definite dates, and may be computed on the amount of the cost after deducting any mills or factories allowances or exceptional depreciation granted in respect of the particular buildings or structures concerned. The expenditure on which annual allowances are granted to persons or firms engaged in developing a source of deposits is usually arrived at by writing down the original capital outlay to a figure referred to as the "residue of expenditure," and this is determined by deducting any allowances which are made, either as initial or annual allowances or as exceptional depreciation granted after a definite date, together with any sum or sums received as sale, compensation or similar moneys in respect of any asset sold, demolished or destroyed.

Allowances authorised by Parliament involve a maze of intricacies and variations, and in order to present a clear and accurate view of the effect of the different provisions of the Acts in regard to this subject, a separate and lengthy volume would be needed; the present object is to emphasise the nature of some of the main items which are not allowed to stand as deductions from profits for the purpose of computing taxable balances, also the nature of certain items which are allowed but are not always claimed.

The total invoiced cost of steel bars acquired for structural alterations or support, including walls, linings, jointing, etc., should be treated in the financial books as a capital charge, the individual amounts being posted direct from the cash book or the purchases journal to the debit side of the asset account already opened in the private ledger. The same applies to additions to mill, factory or works buildings, property extensions and improvements, including headgear in connection with the winning of ore, as well as pipes, fixtures and fittings, etc., and when items of this nature are written off against the metal-producing operations the amounts would have to be added back to the balance of net profit shown in the final account for assessment under Schedule D. A good deal depends upon the kind of substance that is being worked, also on the type of excavation, but the cost of acquiring or installing machinery or tools in accordance with the traditions and customs of the area should be capitalised.

Expenditure representing ground rents, annual interest, annuities and payments of a similar nature from which income tax at the current standard rate

should have been deducted at the time of remittance, is not regarded as a proper charge against profits, and in the case of the one-man business no expenses of a personal nature or which are not directly connected with normal trading should be included on the debit side of the income tax account. All ordinary trade expenses and business charges are, of course, allowed to stand, including the cost of equipment and stores for the use of first-aid parties, protective clothing, and payments made to members of the staff and workers (or their wives or children) who are called up for national service, and within prescribed limits the travelling expenses of weekly wage earners to and from the place of business or operations may be allowed, providing the expenditure has been incurred through circumstances of a specific and unusual nature.

In such matters as motors employed in the handling of ore through various specialised processes, and features such as electric screwdown gear, lubrication neck bearings, etc., it is sometimes difficult to distinguish between maintenance charges and items of expenditure which should be added to the book value of the equipment or debited to a depreciation reserve. Broadly speaking, of course, the guiding principle should be that all amounts representing the cost of new or additional units of machinery or plant, as distinct from the cost of replacing old units, should be treated as capital outlay, and that the cost of acquiring or installing new assets which are intended to replace old equipment, including the cost of restoring plant in such a way as to cancel or substantially reduce the amount which was previously written off under the heading of depreciation, should be dealt with in the accounts as a charge against the depreciation reserve, while all expenditure incurred during the period under review for the purpose of keeping the profit-earning equipment in efficient working order without necessarily prolonging the period of service or useful life of the units, sections, or groups beyond the date assumed in the calculation of depreciation, constitutes a proper charge against revenue and should be included as part of the metal producing operations.

Depreciation

Apart from certain exceptions, the Companies Act requires that in cases where depreciation or replacement of fixed assets is computed by any method other than an amount actually written off or provided for renewals, the actual method adopted in arriving at the figures must be indicated; if no provision has been made for depreciation or renewals, this fact should be stated. Debits of a different nature should not be merged under one general heading or presented in meagre form, for the status of the profit and loss account has now been raised to that of the balance sheet. The amount charged under the heading of Income Tax should state the fiscal year up to which the provision has been made, or it should be made clear that provision has been made on all profits which have been earned to the balancing date; and other taxation, including the Profits Tax, should be shown separately. When a definite amount is retained as provision for a known liability and which, in the opinion of the directors, is in excess of the sum which is reasonably necessary to meet that liability, the excess should be treated as a reserve. Recovery plant accounts should show the figures of scrap for each process, and the next article will indicate methods of recording scrap metal and its recovery.

Pametrada Research Station

The Parsons and Marine Engineering Turbine Research and Development Association



View of design and administrative buildings and laboratories from test house.

This research station has been set up to provide full-scale testing facilities for marine turbine propulsion machinery to meet the maximum requirements for both naval and merchant ships, and to initiate and carry out research for the improvement and development of marine steam and gas turbines and their auxiliary equipment. It was formed, with the support of all British marine turbine manufacturers, to link up with and carry on the work initiated by the late Sir Charles Parsons. In this article is given a brief outline of the activities of the station, with particular reference to some of the metallurgical problems encountered in the development of the marine gas turbine.

THE Parsons and Marine Engineering Turbine Research and Development Association, now more generally known as Pametrada, was formed in May, 1944, to link up with and carry on the work initiated by the late Sir Charles Parsons. It is a collective effort to ensure that British marine turbine engineering remains in the forefront of developments and is supported by all shipbuilding and marine engineering firms who manufacture marine turbines in shipbuilding centres. The acquisition of staff began on September 1st, 1944, when Dr. T. W. F. Brown was appointed Research Director, and by the end of 1944 the staff had been increased to 12, the majority of whom are still with the Association, while to-day it has increased to a total figure of about 180, including about 50 graduates.

A design section was first formed and the Association began designing machinery for the industry on January 1st, 1945. Initially the majority of designs prepared were generally of orthodox Parsons type, usually comprising three-casing reaction turbines driving through single reduction gearing, or compound turbines in which the high-pressure casing comprises a two row wheel followed by reaction blading with a low pressure turbine of all-reaction type. Very early, however, the Association began to develop its own designs, commencing with a high pressure turbine of impulse type employing a solid rotor with discs formed by cutting out from the forging, together with all machined diaphragms of built-up construction.

The first contract for a vessel employing this type of machinery was placed during the summer of 1945. The ship, the *Cortona*, built by Messrs R. and W. Hawthorn Leslie & Co., Ltd., commenced her maiden voyage in

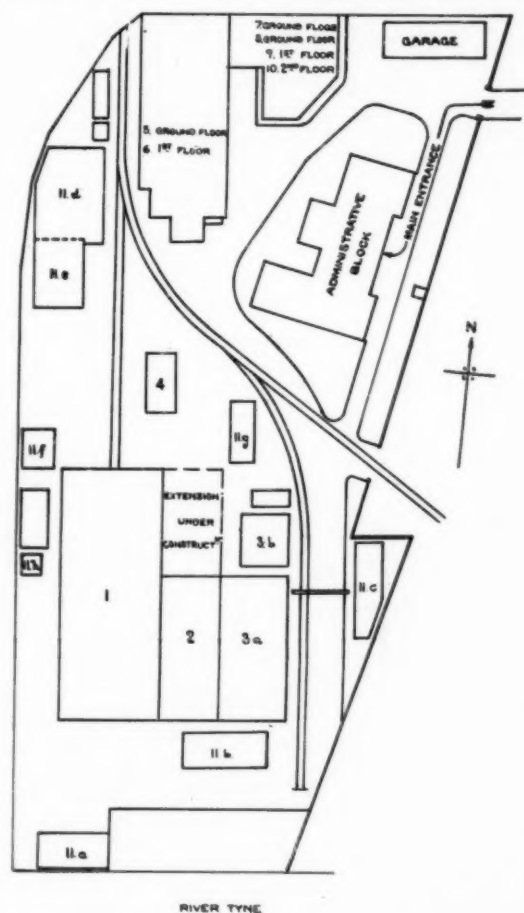
December, 1947, and has been in continuous service since then. Ten ships with machinery of very similar type are now in service and 36 more are under construction. It is noteworthy that the first design enquiry received by the Association on January 2nd, 1945, eventually became a building contract, and the machinery to this design was built by Messrs. Vickers-Armstrongs of Barrow, and installed in the *Surat* and *Shillong*, now in service with the P. and O. Company. Since it was formed the design section has dealt with over 300 enquiries and, while only a fraction of these resulted in building contracts, over 65 installations have been built or are under construction to Pametrada designs. The aggregate horsepower on which royalties have been paid to the Association is over 1,700,000 S.H.P.

One of the important aspects of the design section is that it is associated with the work of member firms throughout their contracts, beginning with the preparation of turbine designs at the tendering stage, revising the design as may be necessary prior to production of constructional drawings, advising during construction and attending the trials of the turbine machinery on completion. In addition, however, it not only suggests the number of researches to be carried out, but enables the results of such researches to be included in machinery building with the minimum expenditure of time.

Testing Facilities

The need has been felt for a testing station in this country at which full-scale tests could be carried out on marine turbine propulsion machinery to meet the maximum requirements for both naval and merchant ships. Consumption trials of marine machinery for both

naval and merchant ships have, of course, been carried out at sea on many occasions, but their value has been limited by the difficulties inherent in sea trials, namely, the difficulties of making accurate measurements of power input, except for vessels with electrical transmission, of separating the auxiliary steam from that taken by the main turbines, and of making long runs under uniform conditions. In turbine machinery trials on shore there has rarely been sufficient boiler and condensing capacity to carry out load trials in the maker's works, and the usual steaming trial prior to installation has been a light-load overspeed test with a duration of overspeed running amounting to minutes. To obtain the accurate performance data which is essential for the development of high efficiency machinery, it is, therefore, necessary to test the sets at full load on shore where the fluctuations caused by the marine conditions are eliminated and where all quantities can be accurately controlled and measured. Testing facilities are provided at this station and testing can be carried out up to a power of 60,000 S.H.P. per shaft.



1. Turbine test house. 2. Research house. 3. Boiler houses.
 5. Gear testing equipment. 6. Sprayers and control blocks and
 equipment for testing. 7. Applied physics laboratory. 8. Gear
 laboratory. 9. Chemical and metallurgical laboratory. 10. Elec-
 trics laboratory. 11. General.

A plan outline showing the arrangement of the buildings.

Arrangement and Functions of the Station

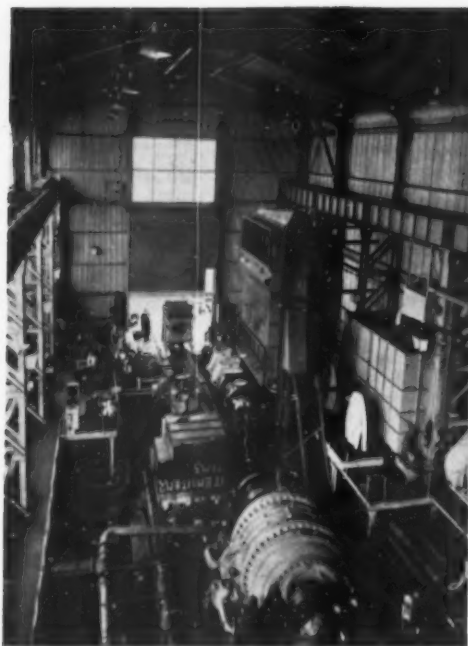
The general arrangement of the station is shown in the accompanying site plan. Extensions are under construction which will absorb all available space on the present site, however, further extensions are contemplated and adjoining land is available for the purpose. The functions of the station are: (1) To initiate and carry out research for the improvement and development of steam and gas turbines, and transmission, for marine propulsion (condensing, feed-heating and re-heating equipment, as forming component parts of the turbine plant, are also included); (2) to establish and equip a test station in which full scale tests of turbine installations may be carried out up to the maximum powers required for warships; (3) to advise in any new design the steam conditions recommended, the layout of the main turbine plant including transmission, the number and dispositions of cylinders, rotor speeds, basic efficiency and to prepare basic designs and preliminary lay-outs; (4) to set up standards of fit and finish required in the principal parts of turbine machinery in order to attain maximum efficiency and reliability in equipment constructed by member firms.

Daring I Machinery Trials

Some idea of the work involved in carrying out tests on marine turbine machinery will be obtained from the following brief summary of the Daring I machinery trials recently completed at the station.

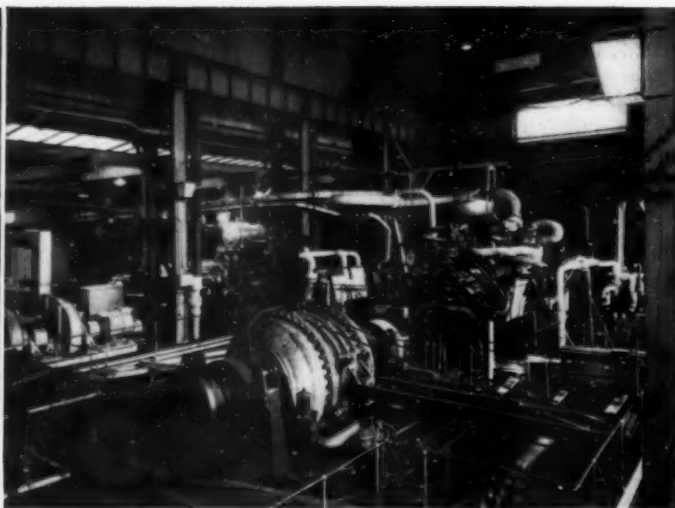
The erection of boiler, turbines and gearing with certain of the associated auxiliaries was carried out by The Wallsend Slipway and Engineering Co., Ltd. The remainder of the work, including the adaptation and installation of research plant and the instrumentation of the machinery under test devolved on Pametrada. The complexity of the instrumentation may be appreciated from the fact that the measurement of no less than 525 separate quantities was involved. Apart from the measurement of temperatures by thermometers, 112 thermocouples were fitted to various points on the installation and continuous records were taken using five automatic multipoint recording instruments. Other readings were taken using potentiometers and indicating instruments. In all, the machinery was run for 407 hours, of which 23 hours comprised astern running. For purposes of description these tests can be divided into two main categories:—(a) the trials proper, mainly concerned with the efficiency and performance of the machinery; (b) associated researches, including the measurement of quantities which are not normally observed.

The trials included the accurate measurement of all important quantities under many different operating conditions. Fuel and condensate were weighed. All relevant steam conditions were observed with the greatest possible accuracy. The output was absorbed and measured by the Heenan and Froude F.A. 18D. dynamometer. Gland steam was measured by means of individual orifices and overall weighing and auxiliary condensate was metered. Steps were taken to measure the spindle leak-off quantities from the manoeuvring valves. The gland condensate was weighed. Air removed from the condenser by the ejectors was recorded on a gas meter. The programme included preliminary proving tests; consumption trials; special vibration and noise trials; manoeuvring trials; and astern trials.



View inside test house showing gas turbine being erected and dynamometer in foreground.

During the course of the trials several associated research schemes were carried out, the more important of which included the following:—(1) In order to assess the distribution of torque between each pair of quill shafts and also to give some idea of power distribution between the turbines, strain gauges were affixed to the quill shafts and connected through slip-rings to a specially developed indicating instrument; (2) Electronic capacitance gauges were fitted to various points on the gearing and turbine shafts to indicate vibrations and movements of the gear wheels and turbine rotors; (3) Measurements were made of pressure drops along main steam piping, across boiler stop valve, and main steam strainer; (4) series of pilot tubes and thermocouples were inserted in the tubes of the main condenser to ascertain the distribution of heat transfer over the condenser heating surface with the ultimate view of effecting an improved design of condenser; (5) Thermocouples were fitted at strategic points in both H.P. cylinder and L.P. inner and outer casings; (6) Three methods of ascertaining the axial position of the aft end of the H.P. rotor relative to the casing whilst running were tried; (7) Brackets were fitted at many points on the H.P. and L.P. turbine with reference points on the gear case and on the test bed for the use of Taylor, Taylor, Hobson alignment telescopes; (8) Rotating thermocouple assemblies were inserted in the bores of both H.P. and L.P. turbines, the leads being led to slip-rings at the forward ends of the rotors; (9) The fan intake noise represents the largest component of the overall machinery noise in this type of plant. Obser-

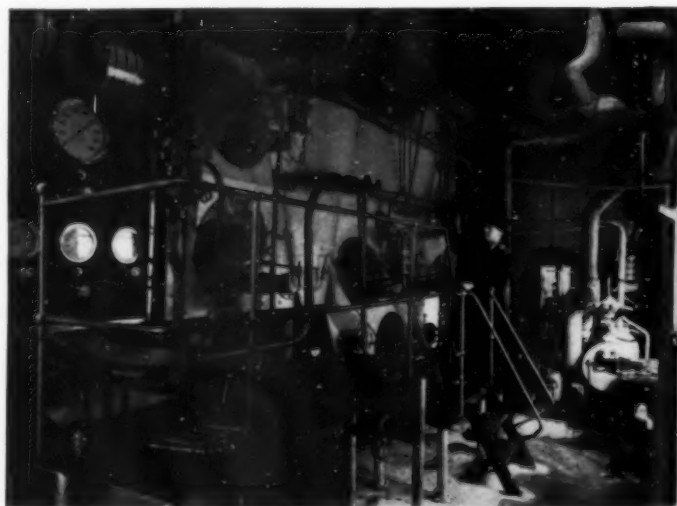


Steam turbine on test with dynamometer in foreground and research house on the left.

vations were made of noise level in the boiler room and fan flats at various powers, and progressive improvements were made in the insulation; (10) Brushes were fitted in order to pick up any electrical voltages generated in the rotors either electrostatically or otherwise.

The Pametrada Marine Gas Turbine

Of particular interest, running on the test bed, was a marine gas turbine which was designed at the station in outline in 1945. The Council of the Association, after studying the project, authorised its construction in January, 1946. Since that date the whole sequence of design, drawing, component development, purchasing, progressing and instrumenting has been undertaken by a part of the Association's staff. Manufacture of the main components was carried out by 11 of the Association's member firms. The complete unit develops 3,500



Front view of high pressure water tube boiler. Normal evaporation rate, 28,000 lb./hr. pressure up to 1,200 lb./sq. in., 950° F. at superheater outlet.



General view of physics laboratory.

h.p. ahead at the normal propeller speed of 85 r.p.m. and also provides ample astern power.

Many aspects of the detail design of this engine are new and there has been no large body of experience to fall back on. In reviewing a few of these factors it should be remembered that the requirements of a marine engine are different from what might at first sight be similar to existing land or aero experience.

On land, large turbine units are housed in buildings suited to the machinery, a comprehensive crane service can generally be provided, auxiliaries can be spread out, and all is made convenient for servicing and cleaning. Marine machinery has, however, to be tailored to fit a steel hull, all the components have to be inserted through the deck and servicing effected when the decks are closed in. The hull also lacks the rigidity of a concrete foundation and each marine engine component has to be considered as part of the working structure. This latter may also be the fate of an aircraft gas turbine but the experiences in this field relate to a highly stressed type of engine, receiving highly skilled maintenance throughout its short life. Its full load running life is approximately 1% of that expected of a marine engine. The creep properties of the high temperature materials used have been accurately known for "full life" tests can be

completed in a few months. In the marine application at least 11 years must elapse from the production of a new material until its "full life" temperature/stress relationships can be determined. Thus the present engine has had to have incorporated in it a few components of limited life. It should be noted, however, that deterioration takes the form of excessive strain and does not occur suddenly. In fact, systematic measurements taken during survey will reveal the number of months reliable "steaming" at known temperatures that remain.

In addition to absolute reliability over a long life, which is the most important factor in the adoption of a gas turbine for marine drives, the question of running costs must be carefully considered in order that this power unit can compare favourably with other successful prime movers.

This involves the question of fuel. At present this gas turbine is running on diesel oil, but, for marine purpose, the cost of such a fuel would be such as to render the unit uneconomic in service in comparison with a steam turbine. From the running costs point of view an obvious possibility is the use of residual oil and this has been tried without solving the general problem. Experimental running at this Research Station has clarified the actual problem and shown that there is no difficulty in burning the heaviest residual oil, but most heavy fuels contain a very small proportion of vanadium compounds. At ordinary temperatures the vanadium is quite harmless, but at normal gas turbine temperatures it produces a sticky compound which clogs the turbine blades. At still higher temperatures this compound becomes completely fluid. It then penetrates even high temperature steels and other metals which are badly



Chemical and metallurgical laboratory



Vickers Projection microscope.

damaged while ceramics soak it up freely but may not suffer severely. Experiments are being carried out at Pametrada to deal with the vanadium content in the oil. Some involve centrifuging and washing the fuel; others are concerned with filtering it from the hot gas stream before it enters the turbine; others again deal with protection of the turbine parts by surface layers of resistant materials. This research may perhaps take some time before an answer is found. The heart of the matter would seem to be neutralising the effects of the corrosion disposed products of combustion, the most intractable of which are vanadium compounds.

Many people, however, are working on the marine gas turbine all over the world, since it has many advantages. Already the efficiency of gas turbine propulsion is greater and the weight less than steam turbine units developing the same power. While occupying considerable volume, the component parts can be assembled in a number of days to suit the space available. The particular cycles employed can be chosen to suit the service requirements of the vessel more easily than with other types of machinery. Gas turbine machinery can be on full load in a matter of minutes, in contrast with steam turbine machinery with which raising steam may take an hour or more.

The Pametrada marine gas turbine, which is now on test at the station, is among the first gas turbines designed for use in a ship and to drive the propeller directly. A hydraulic brake is used to absorb and measure the power which will eventually drive a ship's propeller. When this gas turbine passes its extensive tests it will be installed in a merchant ship. The tests are of great importance; from them will be learnt about gas turbine problems in general and this engine in particular. Measurements are being taken of speeds, pressures and temperatures, casing expansions, fuel consumption and so on. At present, diesel oil is being used but after a while boiler fuel oil will be burnt, though at first only of the lighter types. It is hoped that eventually the gas turbine will burn really heavy fuel oils, which are cheap, but, as mentioned previously, the ash problem is likely to prove one of the greatest difficulties in the development of the marine gas turbine.

It is noteworthy that for more than a year the research staff at this station have been working on a water-cooled turbine intended to be run at temperatures considerably higher than the 650° C. of the present gas turbine. With the help of water-cooling it is expected that the metal surfaces will be at a lower temperature than with the present machine; by this method it is hoped to keep them down to a temperature at which vanadium compounds, present in many residual oils, will neither attack nor stick to the surfaces.

Some Materials Used

Although not by any means the only problem associated with the successful application of gas turbines to marine propulsion the development of materials that will give the needed reliability in service is a very real headache. In the compressor of the Pametrada gas turbine, on which the efficiency of the whole engine is directly dependent, the blades are of aerofoil section and they presented these problems:—(1) they had to withstand corrosion attack from wet salt-laden air; (2) they had to be efficient; (3) they had to withstand high stresses. The choice of material was limited by (1),



The effect of vanadium compounds on a turbine blade*

* Given by H. Roxbee Cox in his recent May Lecture to the Institute of Metals.

and any lowering of stresses of (3) by increasing the blade thickness meant sacrificing efficiency (2).

Following an investigation by Frederick¹ into the properties of drop stamped and die-cast aluminium bronze compressor blades, the rotor blades were precision forged in an aluminium bronze and the stator blades machined from bar of the same material.

The heat exchanger fitted is a compromise between the cross and counter-flow types. The compressed air is directed once through an array of $\frac{3}{4}$ in. dia. tubes while the hot gases pass outside in the counter direction. As a result of tests² an aluminium alloy was chosen for these tubes at a fraction of the cost of stainless steel tubes. The tests carried out in conjunction with the makers revealed satisfactory creep and corrosion resisting properties.

The materials of the combustion chamber lining have to withstand the high temperatures in the flame zone, as well as to secure efficient combustion. The present chambers have their flame tubes or linings in nickel-chrome steel, but refractory linings have also been developed at the same time.

Many problems were encountered in the manufacture of the turbine—notably in connection with the blading and erection procedures and many investigations were carried out. Other investigations conducted related to surface treatments for high temperature joints. It was found that aluminised bolts were least affected by the high temperature. Both the H.P. and L.P. turbines are of double flow design. This was selected since it was desired to have a small diameter for rotor-forgings in austenitic stainless steels and also to have minimum stresses in the blades. The casing is in two parts, the structural strength being provided by a cast stainless steel inner pressure casing.

The choice of materials for the high temperature duties in the turbine was limited. The makers of appropriate materials such as G 18B, Nimonic, and various other alloys assisted—the weakest link in the design being the almost complete absence of creep tests of durations exceeding 10,000 hours. Experiments have also been carried out with refractory materials. This quest for materials capable of withstanding high temperatures, which has been in progress for some years in many countries, is being intensified and many investigations are in progress although little or no reliable data is available concerning the further developments in this field. The high molybdenum, chromium and tungsten

¹ S. H. Frederick, Pametrada Report No. 45.

² S. H. Frederick, Pametrada Report No. 36.

alloys under development are not expected to extend the practical temperature limit beyond 960° C., although liquid cooling seems to offer distinct possibilities for certain applications. Many of the metals that might be usefully employed are not readily available and in consequence more attention has been directed to ceramics which resist oxidation and have fairly good strength at high temperatures, they have, however, little resistance to thermal shock and this fact has led to the development

of metal-refractory combinations possessing good resistance to thermal shock, and many are at work on different materials which have both metallic and non-metallic properties; powder metallurgy methods are being employed. Surprising results may be expected from the work in progress, but tests of long duration will still be necessary to enable an accurate appraisal of possible application of these results to the marine gas turbine.

Staff Changes and Appointments

MR. J. EDMUND GAMAGE and MR. DONALD L. CAMPBELL have been appointed joint sales managers for the Electric Furnace Co., Ltd.

SIR WILLIAM A. STANIER, F.R.S., has been appointed Chairman of the Board of Directors of Power Jets (Research and Development), Ltd., by the Ministry of Supply. He has been a Director of Power Jets since 1944. Sir William, who is a Past President of the Institution of Mechanical Engineers, is also Chairman of the Ministry of Supply Industrial Gas Turbine Committee and the Mechanical Industries Division and the Welding Committee of the British Standards Institution. He is a member of the Executive Committee of the National Physical Laboratory and of the Council of the B.S.I.; a Director of the Fulmer Research Institute and a Governor of the Aeronautical College.

MR. M. R. NEVILLE, M.C., M.A., A.M.I.E.E., has been appointed Manager of the Publicity Organisation of the General Electric Co., Ltd. to succeed Mr. C. Pinkham, who is retiring at the end of this month. Mr. Neville served in the R.G.A. in the first world war, and joined the General Electric Co. in 1919 under Lord Hirst's ex-officer training scheme. In 1924 he joined the Sales Department and was appointed Assistant Sales Manager in 1933.

MR. JAMES VENUS has taken up the appointment of Naval Architect with the Aluminium Development Association in succession to Mr. G. L. Watkins, who resigned to join another organisation. Mr. Venus served his apprenticeship with Messrs. R. & W. Hawthorn Leslie & Co., Ltd., and has held positions with several companies, both on the commercial side and in the shipyard and was, for some time, on the staff of the Ministry of Transport as a surveyor.

MR. HUGH CAMERON has been appointed Manager of the Sheffield District Sales Office of the United Steel Companies, Ltd., in succession to the late Mr. J. P. Ibbotson.

MR. J. H. GROOCECK, B.Sc., has been appointed Chief Electrical Engineer for the Abbey, Margam & Port Talbot Works of The Steel Company of Wales, Ltd. On obtaining his degree, Mr. Grooceck joined Messrs. Sulzer Bros., Ltd. at Winterthur, Switzerland; in 1934 he took up a post with Imperial Chemical Industries, Ltd. and later became Regional Electrical Engineer for the South Wales factories of the Metal Division.

MR. G. DUNN, MR. W. HAIGH, J.P., MR. W. JOHNSTONE, SIR WILLIAM PALMER, K.B.E., C.B., and MR. A. WHITAKER have been appointed members of the Council of Industrial Design by the President of the Board of Trade, who also re-appointed SIR LEIGH AHTON, MR. NOEL CARRINGTON, MR. G. W. LACEY, C.B.E., B.Sc., MAJOR F. J. STRATTON, C.B.E., SIR

CHARLES B. L. TENNYSON, C.M.G., and DR. W. J. WORBOYS to be members of the Council on the termination of their existing appointments. MR. ANDREW NAIRN and MR. R. LYON SCOTT have been re-appointed to the Scottish Committee.

MR. J. F. R. JONES, Chief Constructional Engineer at Messrs. John Summers & Sons, Ltd., has succeeded Mr. E. T. Judge as Chairman of the Plant Engineering Panel of the British Iron & Steel Research Association.

MR. S. R. HOWES has been appointed General Manager of Samuel Fox & Co., Ltd., one of the four main branches of the United Steel Companies, Ltd. Mr. Howes, who succeeds the late Mr. S. A. Jackson, joined the Company in 1935 as Works Manager, Light Department, and was appointed General Works Manager in 1944, becoming a Director in 1942. Further appointments include: MR. H. P. FORDER to be Assistant General Manager (Commercial and Administration); MR. O. INMAN, Director and Chief Engineer, will, as the senior resident director, deputise for Mr. Howes when necessary; MR. J. DAVID JOY to be General Works Manager; MR. G. THICKETT, Works Manager (Heavy Departments); and MR. H. D. SAWTELL, Works Manager (Light Department).

MR. G. R. BOLSOVER, Assoc. Met., F.Inst.P., F.I.M., who joined Samuel Fox & Co., Ltd. in 1924 as Chief of Research and was appointed Chief Metallurgist in 1925 and a Director in 1936, will relinquish his executive duties at the end of the present month and will be appointed Consulting Metallurgist to the Company; he will remain a Director.

MR. W. J. NICHOLLS, B.Sc., M. Inst.C.E., A.M.I.E.E., has joined the staff of Aluminium Wire & Cable Co., Ltd. as Assistant to the Chief Engineer. He resigned his position as Overhead Lines Design Specialist at the headquarters of the British Electricity Authority to take up this new appointment.

MR. F. J. WYMER, C.B.E., General Assistant to the Chief Regional Officer, Southern Region, has been appointed Assistant Chief Regional Officer, Southern Region, Waterloo.

MR. T. S. KILPATRICK has been appointed a Director of the Workington Iron & Steel Company, a branch of the United Steel Companies, Ltd. Mr. Kilpatrick became Commercial Manager at Workington in 1948.

MR. W. O. GASCOIGNE, 28, Priory Road, Kenilworth, Warwickshire, has been appointed Resident Engineer in the Midland Area for Renfrew Foundries, Ltd.

Change of Address

BORAX CONSOLIDATED, LTD., advise that as from June 26th, 1950, they will be re-occupying their pre-war offices, which were requisitioned during the war, at Regis House, King William Street, London, E.C.4.,

Reviews of Current Literature

THERMODYNAMICS OF DILUTE AQUEOUS SOLUTIONS

By M. J. N. Pourbaix (translated by J. N. Agar), pp. XI + 136 (Edward Arnold & Co., London, 1949), 30s. net.

This interesting little book, which has been well translated, deals with the thermodynamics of aqueous solutions in a clear and simple manner. The treatment is developed in terms of chemical potentials and particular attention is paid to the graphical representation of equilibria in such solutions.

The book opens with a short introduction and a classification of reactions according to whether or not they involve hydrogen ions, or electrons, or both. There follows a short statement of the laws of chemical and electrochemical equilibrium expressed in terms of the chemical potentials of the participating substances or ions, and of the electrical potential, if the transfer of electrons is involved. Examples are given of typical reactions and of graphical methods of representing the equilibria. The results strictly represent the relationships between the activities of the participating substances or ions, the pH and the electrical potential. To use the activities as concentrations is only permissible at very low concentrations, so that the results are not of such immediate and wide application as might at first be supposed. The findings, and particularly the diagrams, are nevertheless of great interest and extremely useful. The nature of the diagrams also is such that a vast amount of experimental work can be summarised in them and can be readily appreciated and indeed remembered by eye. When used to summarise data relating to complex systems, the diagrams permit the important equilibria to be separated immediately from the unimportant, so that the former can then be considered more fully in terms of concentrations and activity coefficients. Such diagrams should be of great value to those concerned with industrial and analytical problems, particularly in cases where very accurate information is not required for the decisions which may have to be made.

Since this book should have a wide appeal, it seems unfortunate that the section dealing with the laws of chemical and electrochemical equilibrium in terms of chemical potentials should not make any attempt to explain the latter. Two references are indeed given—though both are to foreign papers—and standard textbooks are admittedly available. It is felt, however, that since many persons unfamiliar with the concept of chemical potential should find the book of value, a chapter of explanation would have been amply justified in this English version.

The latter part of the book deals in detail with the systems $\text{Cu-H}_2\text{O}$, $\text{Fe-H}_2\text{O}$, $\text{Cr-H}_2\text{O}$ and $\text{N-H}_2\text{O}$. The first three of these are of great interest to that concerned with corrosion, particularly on account of the potential-pH diagrams on which three "corrosion domains" are delineated. The domain of *corrosion* covers the conditions in which the metal is attacked; the domain of *immunity* covers those in which the potential of the metal is depressed too far for passage into the combined state, so that no attack can take place; the third domain of *passivation* covers conditions in which an almost insoluble corrosion product can be formed which may be able to stifle further attack. The diagrams thus

provide the thermodynamic background from which detailed explanations of corrosion phenomena may be developed and with which they should accord.

The book concludes with an appendix listing the standard chemical potentials of inorganic substances relative to the pure elements. These are the values which have been used for the calculations and diagrams throughout the book, and it is a little unfortunate that they have merely been taken from standard tables. Although it would perhaps be expecting too much in too small a compass, the value of the book would have been greatly enhanced had the original data been critically reviewed and likely accuracies been proposed either for the curves in the diagrams or for the standard chemical potentials. The book is reasonably well printed and the diagrams are clear.

F. D. RICHARDSON

ENGINEERING STRUCTURES.

Volume 11 of the Colston Papers, presenting contributions to the Symposium on Engineering Structures held in September, 1949, in the University of Bristol under the auspices of the Colston Research Society. Published as a Special Supplement to *Research* (1949), 49, pp. 260, 21 figs. Butterworth's Scientific Publications, 25s. (by post 1s. extra).

THIS symposium will be of interest to many who are not specialists in the design of structures. Engineering structures, such as aircraft, bridges and ships, absorb large quantities of metal and the discussions provide much incidental information on the nature of this demand and on the suitability of the available materials in various applications.

The book opens with a review by Sir Richard Southwell of current trends in structures research. He cites some interesting cases of how the introduction of new materials or methods may lead to factors previously ignored in design becoming important. Many subsequent contributions deal with the correlation of calculation with observations of the strength and stiffness of beams, plates, struts, etc., fabricated from the popular structural materials such as the steels and the aluminium alloys. Behaviour in the plastic range receives a great deal of attention. It is recognised that if the ultimate carrying capacity of a structure can be estimated accurately a small amount of yielding may be allowed even at working loads, providing, of course, that the factor of safety over the collapse load is maintained and the deflection is not excessive. This places a new emphasis on ductility as a desirable property of the material.

New studies of members made from thin plates bent to form channel, zed and top hat sections are reported. This work is of special interest at the moment because thin plate construction may be adopted more widely for light building frameworks as supplies of steel sheet improve. A final paper on structural testing (J. B. B. Owen) includes descriptions of service failures and of methods of shock and endurance testing that will interest those responsible for the specification of materials.

The symposium was by no means exclusively domestic. Authorities from other European countries and from America attended. The discussion printed after each paper frequently forms a cross-section of international opinion on the questions at issue. Consequently the book provides a unique, up-to-date review of the progress of research in engineering structures.

R. M. HAYTHORNTHWAITHE.

The Use of Oxygen in Steelmaking

Recent British Developments with Electric Furnaces

After three years of trials and development, two British firms are now regularly using gaseous oxygen in the refining stage of steel production in the electric arc furnace. The Brymbo Steelworks, Ltd., are operating on 30-ton furnaces making low alloy steels whilst Edgar Allen & Co., Ltd., are doing what was previously virtually impossible—making stainless steel by remelting stainless scrap in the arc furnace.



Courtesy of Brymbo Steelworks Ltd.

The blow in progress.

THE post-war years have seen a marked awakening of interest, both in Europe and America, in the use of gaseous oxygen in the various processes operated in the iron and steel industry. It is true that the idea had received consideration at an earlier date—the use of an oxygen enriched blast for the production of iron and ferro-alloy, for example, having been tried by Soviet technicians some years before the war—but the conditions prevailing after the cessation of hostilities provided a new impetus to the study of the problems involved, the post-war demand for steel emphasising the need to explore every avenue which might lead to an increase in production from the capacity available.

The various steelmaking processes, open-hearth, electric-arc and converter have been investigated and it has been shown that, under certain circumstances, all can utilise gaseous oxygen with beneficial results, whilst, to a lesser extent, there is evidence that iron and ferro-alloy making may also derive some benefit.

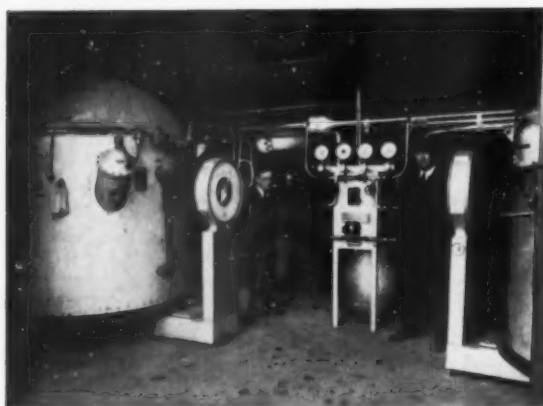
Cost of Oxygen

Without doubt, one of the major factors influencing the widespread application of gaseous oxygen is its cost. Although the raw material—air—is one of the few commodities which have not rocketed in price in recent years, the selling price has to cover the capital cost of the plant, together with such running costs as labour and power, and the cost of such consumable stores as drying agents and lubricants. At present, in this country, the oxygen used in the iron and steel industry is of high purity, and is supplied in cylinders or in liquid-oxygen bulk-quantity containers from which it is transferred to

an evaporation plant at the consumer's works. For many iron and steelmaking purposes, such a high purity oxygen is unnecessary and a possible future development is the establishment, at the steelworks, of "tonnage oxygen" plants producing oxygen of 90-95% purity continuously. Such plants are based on the same principle as the high-purity plants, i.e., the difference in boiling point of liquid oxygen and liquid nitrogen. In the medium-purity plants, however, the liquid oxygen is immediately evaporated and its "negative heat" is recovered, the efficiency being thereby improved. A further advantage is to be seen in the elimination of the cost of transport. On the other hand, for a "tonnage plant" to operate at maximum efficiency, a continuous output of at least 50 tons of oxygen per day is necessary, for which there must be a corresponding continuous demand. As an intermediate course, some reduction in cost would no doubt result if the building of larger oxygen plants on the present lines were to be justified by a substantial increase in demand.

The Blast Furnace

The only process in which a continuous demand for large quantities of oxygen is likely to arise is the production of iron in the blast furnace, where oxygen enrichment of the blast would speed up production and reduce coke consumption. On the other hand various factors limit the amount of oxygen which can be carried in the enriched blast to a figure below 30% and it is doubtful whether any appreciable saving would be effected thereby. For smelting normal ores, therefore, the probability of utilising oxygen-enriched blast seems



Courtesy of Brymbo Steelworks, Ltd.

Oxygen Evaporation Plant.

rather remote, while for low grade ores, the adoption of efficient beneficiation treatment may prove to be a more economical proposition.

The Open-Hearth Furnace

Oxygen has been successfully applied in the open hearth process for melting down and for refining. As an aid to quick melting an oxygen-enriched flame is utilised, the oxygen being introduced just beneath the base of the flame. This procedure is ideal for 100% cold scrap charges, where the surplus heat can be taken up by the charge without overheating the roof, but charges comprising large amounts of "mixer metal" cannot derive much benefit from the use of an oxygen-enriched flame.

Rapid removal of carbon, combined with a saving of fuel, can be achieved by the introduction of oxygen to the bath at the refining stage. In this case the gas enters the bath by way of a lance, i.e., a long steel tube which is slowly consumed in the process. The oxygen replaces part or all of the ore and mill scale normally fed at this stage, and the oxidation of various elements results in a rise in bath temperature and a reduction in fuel consumption. The agitation produced by the introduction of the oxygen causes a better mixing of slag and metal in the oxidising atmosphere present in the furnace and helps to speed up carbon removal still further. American experiments on this point have shown that even nitrogen will speed up carbon removal whilst compressed air, which is considerably cheaper than "tonnage oxygen," is only slightly less than half as efficient. It will be seen, therefore, that there are many points to be considered before a decision can be reached on the value of oxygen in the refining of steel in the open hearth furnace.

The Converter

When we come to consider the converter, we are on safer ground in forecasting that enrichment of the air blast by oxygen is likely to find increasing application in the near future. Oxygen-enriched converters have been operated successfully for some time in Germany, and during recent years a side-blown converter, using enriched blast has been operated regularly in the steel foundry of Messrs Catton & Co., Ltd., at Leeds. The effect of the enrichment is to enable hotter steel to be

produced by a shorter blow, to enable lower silicon or phosphorus irons to be used, or to enable a larger percentage of scrap to be charged. The advantage of rapid blows and hotter metal in the steel foundry will be obvious, particularly where thin sections have to be run. Lining life considerations limit the permissible oxygen content to 30-35%.

The Electric Arc Furnace

Early in May a confidential conference of steelworks technicians drawn from member firms of the British Iron and Steel Research Association was held at Ashorne Hill, Leamington Spa, to discuss the most recent British developments in the use of oxygen in steelmaking in the electric arc furnace. For some time now, oxygen refining has been the regular practice at the Brymbo Steelworks, Ltd., near Wrexham, and at Edgar Allen & Co., Ltd., Sheffield. The developments are the work of the individual firms, although the Research Association has supplied a certain amount of background knowledge and technical assistance. After three years of experiments and trials, it was felt that the technique had reached the stage where it would be valuable to share the knowledge acquired by the pioneering firms and further developments in the works of several manufacturers are expected.

Developments at Brymbo

Brymbo Steelworks has a long and varied history, many of the famous ironmasters having been associated with it at one time or another. To-day it presents a curious mixture of past and present; on the one hand, the visitor can see the remains of one of the old stone-built blast furnaces which was erected in the 1790's and made iron until the 1890's, and on the other hand, there is an arc furnace plant, comprising three 30-ton basic furnaces, on which recent developments in the use of oxygen have taken place. The plant also includes a blast furnace, producing 1,000 tons of iron per week; a basic open-hearth steel plant, comprising four furnaces of 40, 60, 60 and 90 ton capacities, which absorb most of the blast furnace output as hot metal; and a bar mill.

When, for security reasons, it was considered inadvisable to have such a high proportion of the country's electric steel melting capacity concentrated in Sheffield, Brymbo was chosen as a dispersal site and an electric melting shop was erected, equipped with three 15-ton furnaces which have since been re-designed at the works to increase the capacity to 30 tons.

The output of the Brymbo arc furnace plant consists chiefly of low alloy steel and silicon steel suitable for the manufacture of sheet for use in the construction of electrical machines, where specific magnetic properties are required. In order to meet these requirements, low carbon contents, of the order of 0.05%, are required, a type of steel for which oxygen injection is particularly suitable. The advantages resulting from the introduction of oxygen-lancing on this plant are: (a) a saving of electrical power, and (b) a speed-up of the refining stage.

The charge consists of three 10-ton skips of cold scrap, no hot metal being used, and the operation of the furnace proceeds normally until the bath is completely melted and ready for the refining stage to begin. At this point, with a bath temperature of the order of 1,500° C., the power is switched off and the electrodes are raised well clear of the molten slag. Then, instead of feeding ore and mill scale, a steel-plate shield is placed in front of the furnace door and an oxygen lance, a $\frac{3}{4}$ in. bore steel

tube connected to the oxygen supply, is inserted into the bath at the slag/metal interface. The flow of oxygen through the tube cools it sufficiently to slow down the rate of wastage of the tube to reasonable proportions. To compensate for the burning away of the end of the tube the operator feeds it into the bath at such a rate as to keep the tube end at the slag metal interface.

At the commencement of the blow, as the FeO content of the bath is rising, dense brown fumes are evolved which, as the carbon starts to burn off, give place to flames. So long as carbon is being burnt off at a reasonable rate, the flames persist, but towards the end of the blow, when the rate of carbon removal has fallen, the FeO content of the slag rises again and the flames subside. If possible, the carbon is run down from the melt to the desired composition in one operation, but intermediate slagging is necessary in some circumstances. Following the blow the heat is finished in the normal manner with the power on again.

It is interesting to note that in the electric arc furnace, where the atmosphere is less oxidising than in the open hearth furnace, compressed air cannot be used successfully for carbon removal.

During the blow, such elements as manganese, silicon and phosphorous are also oxidised and the net result of all these reactions is a rise in the bath temperature from about 1,500° C. to 1,650° C. Care has to be exercised in controlling the rate of flow of oxygen, as the violence of the carbon oxidation may cause the metal to boil over the sill. When the procedure was first introduced, the melters were not over enthusiastic, but now they have had some 18 months experience of the method they have no desire to return to the more arduous business of feeding ore and scale during the refining stage.

In the early stages of the blow, the rate of carbon removal is much faster than it is towards the end, and although it is comparatively easy to attain figures of 0.05% C., further reduction is more difficult and the FeO content of the slag rises to such a value that excessive slag-line attack of the dolomite lining occurs. Otherwise, no ill effects on the lining, which is made by ramming dry dolomite with a pointed rammer, have been experienced as a result of oxygen blowing.

Daily deliveries of liquid oxygen, amounting to 94,000 cu. ft. (at N.T.P.) are transferred to two liquid oxygen evaporators which are located on weighing machines calibrated in cubic ft. The pressure of gas in the evaporator house is 250 lb./sq. in. but by the time the end of the lance is reached this has probably fallen to something nearer 100 lb./sq. in.

Although the saving in power will vary from charge to charge, a figure of 125 kWh/ton resulting from the use of 350-400 cu. ft. per ton may be taken as representative.

Some idea of the rate of carbon removal can be gained from the fact that a typical heat, melting 0.49% C. was reduced to 0.05% C. in about 25 minutes.

It is not inappropriate that Brymbo should be a pioneer in the use of oxygen in the arc furnace, as it was there that an earlier use of oxygen—for opening-up hard blast furnace tapholes—was first introduced into this country from Germany.

The Remelting of Stainless Steel Scrap

At the works of Messrs. Edgar Allen & Co., Ltd., in Sheffield, resort to the use of the oxygen lance in the arc furnace was made for a totally different reason. Prior to this development, most of the stainless steel scrap

was almost unusable by firms manufacturing stainless steel in arc furnaces. Manufacturers in this country were faced with an ever increasing tonnage of scrap stainless material, containing valuable elements such as nickel and chromium, which could only be remelted in comparatively small quantities in high frequency furnaces.

One element in stainless steel which must be under control within a narrow range is carbon, and in the austenitic stainless steels it is often desirable to finish with a carbon content less than 0.08%. In the electric arc furnace, due to the close proximity of the carbon electrode to the metallic charge, experience has shown that it is impossible to prevent a pick-up of carbon the steel during melting, often to such extent as to render the steel practically unusable. Formerly, a relatively slow method of carbon removal had to be used, in the course of which chromium was also removed. It was this feature which very severely limited the quantity of stainless scrap in the charge. In the new technique, employing oxygen, it is immaterial whether the carbon in the molten charge is high or otherwise, because the excess carbon can readily be removed. From the practical point of view, however, a high carbon content (of the order of 0.5%) is preferable before blowing is commenced, and pig iron is sometimes charged for this purpose after the melting sample has been analysed.

Charges consisting entirely of stainless steel scrap are melted in the furnace with no regard for carbon contamination. When molten, oxygen is blown into the metal and oxidises such elements as silicon and manganese, the temperature of the bath being raised as a result. Subsequently carbon is burnt out until, finally, a figure as low as 0.05% C. may be reached, and this without nearly the same loss of chromium. During the



Courtesy of Edgar Allen & Co., Ltd.

Melter about to lance oxygen into electric arc furnace. The shop superintendent who controls the process is seen on the right looking through the side door of the furnace.



Courtesy of Edgar Allen & Co., Ltd.

Oxygen lancing operation in progress on a charge of stainless steel.

oxygen injection some oxidation of chromium does take place, thereby generating more heat, and the oxide passes into the slag. At a later stage in the process, addition of ferro-silicon causes the chromium to pass back into the molten steel to the extent of 90-95%. Addition of ferro-chrome is also usually necessary in order to adjust the chromium content to that called for in the specification. Recently, very successful experiments have been carried out using silicon-chrome, an iron-chromium-silicon alloy produced as an intermediate product in the manufacture of ferro-chrome. With silicon-chrome additions, the figures for chromium recovery have shown a slight but definite improvement over those obtained using ferro-silicon and ferro-chrome.

The furnace in which the oxygen injection process has been applied to the melting of stainless steel scrap has a capacity of 5 tons and, as is the case at Brymbo, the power is switched off and the electrodes raised at the commencement of the blow. A considerable amount of heat is produced during the blow, and although no means have yet been developed to measure the bath temperature at the end of the blow, it is believed to exceed 2,000° C. It is the high temperature attained which is responsible for the success of the process, since with increasing temperature carbon tends to be oxidised in preference to chromium. The blowing period is normally less than 10 minutes, in all, and the reaction has to be carefully controlled by interrupting the flow of oxygen from time to time. The temperature rise during the blow is such that, unlike the practice at Brymbo, the power is never put on again, and it is usual to have to wait an hour and a half before the metal is down to casting temperature. During this period the finishing operations are carried out as outlined above.

As would be expected, the high temperatures involved make severe demands on the refractory materials used. In this connection the thick slag covering the bath during the blowing operation is beneficial in protecting

the roof from intense radiation and this has no doubt contributed to the useful roof life attained. For the hearth, dolomite is used with reasonable success, but it is felt that a magnesite hearth would be an improvement. The difficulty in this case arises in the fettling of eroded regions, but it is hoped that further work will result in a solution of this problem.

As a guide to the sort of results obtained in the process, figures taken from the records of some half-dozen heats have been averaged. From these it appears that a carbon content of 0.48% (melting sample) can be reduced to 0.07% in 9 minutes, by the use of 3,740 cu. ft., of oxygen.

Apart from the fact that the process is the only means of utilising stainless scrap in the electric arc furnace, the low carbon contents obtainable reduce the amounts of titanium and niobium necessary to ensure freedom from the "weld-decay" type of failure. The attainment of such low carbon contents as 0.02% or 0.03% whereby it might be possible to eliminate the need for stabilising elements entirely, again results in excessive attack on the lining.

This new technique is undoubtedly the answer to this country's remelting of many thousands of tons of stainless steel scrap. Moreover, it is claimed that the quality of steel produced is, if anything, superior. It is no idle claim, therefore, that this is one of the most important developments in the history of stainless steel.

Metals in the Service of Mankind

An exhibition under the above title will be open at the Science Museum, South Kensington, from July 7th to September 30th, 1950, which will illustrate, by the aid of charts, photographs, samples, apparatus and models, the story of the extraction, refining, fabrication, testing and uses of the common industrial metals, the rarer metals and the precious metals. Sections will be devoted to illustrate processes such as welding, metal surface finishing and electrodeposition. Admission is free.

An Exhibition Handbook containing a guide to the exhibits together with an authentic and readable "Story of the Metals" can be obtained from the Museum or from the Secretary, The Institution of Metallurgists, 4, Grosvenor Gardens, London, S.W.1, at a cost of 1s. (by post 1s. 2d.).

The Achema Convention

THE ACHEMA IX Exhibition and Convention for Chemical Apparatus, which will be held in Frankfurt-on-Main from July 9th to 16th, is more than fully occupied. No fewer than 450 firms—10% more than in 1937, when the last ACHEMA was held, have booked over 10,500 sq. metres (11,300 sq. ft.) net space in eight large halls, in order to show their latest designs in chemical apparatus, machinery and auxiliary equipment. The lively interest displayed in this Exhibition and Convention, which is being held again for the first time after an interval of 12 years and is unique of its kind for Europe is shown by the fact that over 2,000 personal applications for tickets arrived within four weeks after the invitation to visitors had been sent out. The visit of some four or five thousand experts in chemical science and technics may be reckoned with. All those attending the Convention are sent a copy of the ACHEMA Year Book, 1940/50 free of charge. It is a volume of 700 pages and is intended as preparation for the visit. Write to the DECHEMA, 10 Ulmenstrabe, Frankfurt-on-Main.

Recent Developments in Materials, Tools and Equipment

A New Machine for Charging Sheet Annealing Furnaces

BATCH furnaces are widely used for box annealing piled steel sheets. In this type the sheets are usually placed on a base and covered to form a heavy box which is luted at the junction with the base by a sand seal. The annealing boxes, which vary in their capacity, are pushed into the furnace by various means, usually on ball races or bogies by a charging machine and, after heating and soaking for a required period, are withdrawn to cool. Various methods have been developed with a view to speeding up the operation and in this connection a new charging machine, developed to service a battery of furnaces, is noteworthy. This machine, designed and constructed by The Wellman Smith Owen Engineering Corporation, Ltd., has recently been placed in service in the sheet mill annealing department of Messrs. John Summers & Sons, Ltd., at Shotton Steel Works. It accommodates two boxes of sheets, each weighing 53 tons, for which the furnaces are designed; the boxes being received from racks situated on the outer side of the runway and, after annealing, they are returned to the racks for cooling.

The track on which the machine travels is laid at floor level, and consists of two 6 in. sq. bars spaced at 19 ft. centres running parallel with the shop. The annealing furnaces are on one side of the track and the cooling racks on the opposite side. Kick rollers bearing on rails attached to the walls are placed at both ends of the machine to take up end thrust.

The charging machine, which has a total weight of about 76 tons, consists of a travelling undercarriage on which four heavy rails are placed at right angles to the main track. These can be brought into alignment with similar rails in the furnaces and on the cooling racks, leaving a gap of 2 in. between the ends. The undercarriage, which is of welded construction, is mounted on four steel tyred wheels, two of these being driven by a 40 h.p. electric motor through spur reduction gear and a cross shaft. A travelling speed of about 100 ft. per min. is obtainable.

Mounted side by side on the four rails are two long narrow traversing carriages each consisting of a lower member or racking frame and a cast steel hoisting frame. Each racking frame, which is simply a steel slab, is carried on six axles fitted in gunmetal bushes inserted in the frame. A supporting wheel is keyed to each end of the axles and between the frame and each wheel a narrow roller smaller in diameter is placed and is free to rotate on the axle. These rollers carry the hoisting frames, which are actually two long inverted troughs having their supporting edges machined in the form of a series of inclined planes. The upper and lower ends of



A general view of the charger fully loaded prior to charging.

these surfaces are shaped so that the hoisting frames will remain stationary on the rollers when in either the hoisted or lowered position.

A machine cut steel rack bolted underneath each racking frame enables the complete carriage to be traversed into the furnace or out to the cooling racks. When traversing there is no relative movement between hoisting frames and racking frames.

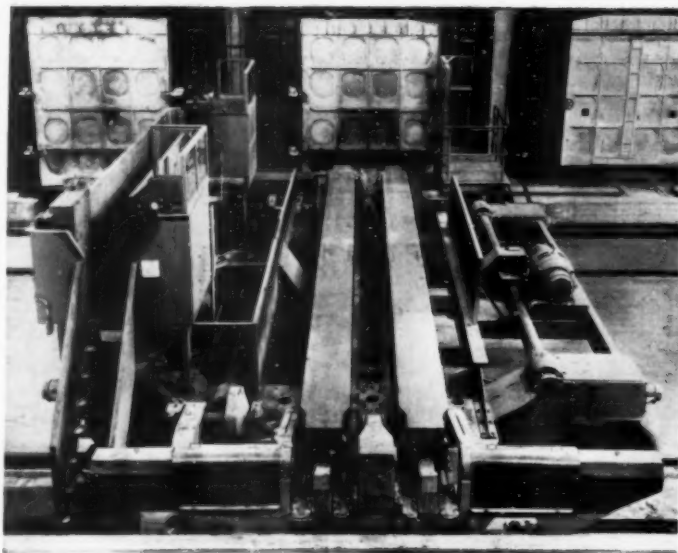
Close to each end of the main undercarriage are two pinions which drive the racks, and they are driven at the same speed by means of bevel gears from a connecting shaft situated on the opposite side of the machine to the travel gear. This connecting shaft is coupled to a two-speed gearbox driven by a 55 h.p. electric motor. A slip coupling is provided between the motor and gearbox and traversing speeds of 5 ft. or 30 ft. per min., according to the gears engaged, are obtained, which gives a useful range for manipulation.

Four horizontal sliding stop pins are carried in heavy brackets disposed at the four corners of the undercarriage. Holes near the end of the hoisting frames enable the pins to be passed individually through each frame and to engage another bracket fixed between them. The holes are elongated to allow vertical movement of the hoisting frames with the pins inserted. The pins which are fitted with rollers mounted on needle bearings are actuated by means of a double acting hydraulic cylinder. Oil pressure is supplied by a pump direct coupled to a 4 h.p. electric motor which can only be started when the gear change lever is in the slow speed position.

As the machine is more than 30 ft. wide, it is provided with a control stand at each end with a duplicate set of controllers, valves and speed gear change levers to facilitate alignment with furnaces and cooling racks.

When correctly aligned at the cooling side of track the traversing carriages are run out in the lowered position and pass underneath the charging boxes which are supported on built-up rails and brickwork. The holes in the hoisting frames are then brought into alignment with the stop pins, the slow speed gear engaged and the hydraulic pump started. The stop pins are inserted by opening the control valve, thus preventing horizontal movement of the hoisting frames. When the traversing motor is re-started the racking frames are moved a distance of 21 in. causing the hoisting frames to lift $3\frac{1}{4}$ in. vertically as the rollers move underneath their inclined lower edges. This is sufficient to lift the boxes clear of the fixed supports and the stop pins are then reversed until clear of the hoisting frames. The high speed traversing gear is then engaged and the two carriages traversed back to the machine carrying the boxes on the hoisting frames.

After travelling the machine into position opposite the furnace to be charged, the furnace door is raised and the carriage is traversed on to the furnace hearth rails. The slow speed gear is then engaged, and the stop pins inserted in the holes in the hoisting frames, allowing the racking frames to be again moved 21 in. and lowering the hoisting frames. The boxes are deposited on packing



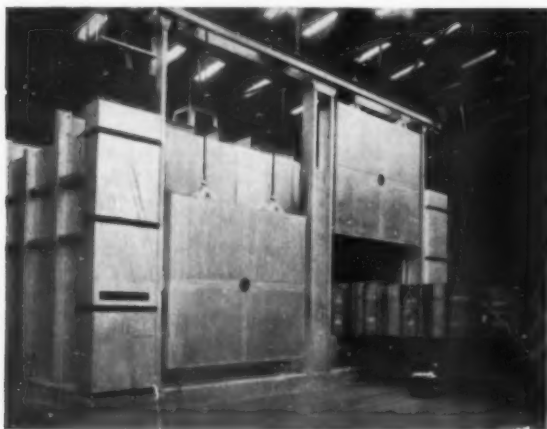
The general arrangement of the Wellman 106-ton charging machine showing the rocking frames.

pieces supported by brickwork on either side of the furnace rails, and after withdrawing the stop pins the carriages are withdrawn to their travelling position on the machine.

All gears, with the exception of the rack pinions, are totally enclosed and suitable heat shields are fitted to protect the carriage wheels.

Twin Chamber Coil Annealing Furnace

RECENTLY put into commission at the British Aluminium Falkirk Rolling Mills, is a twin chamber coil annealing furnace for aluminium and aluminium alloys. Manufactured by G.W.B. Electric Furnaces, Ltd., this furnace incorporates a development in design which gives a higher working efficiency than the normal electric furnace which utilises slow speed air turbulence



A new twin chamber coil annealing furnace incorporating a directional forced air circulation system.

to impart heat to the charge. This superior performance is due to the G.W.B. directional forced air circulation system, which not only ensures rapid initial heating, but also a maximum degree of thermal uniformity within the working space. By this system the heated air is driven at a speed of 10-15 ft./sec. through the charge coils, back into the multiblade centrifugal fans for recirculation over the heating elements situated in the specially designed ducting under the arched roof. The high speed forced convective currents intimately contact all faces of the charge, giving the highest degree of regular heat-transfer. In addition to directional air flow, the baffle plates provided prevent uneven direct radiation from elements to charge, avoiding the danger of "hot spots." It is a feature of the design that the heavy nickel chromium strip elements are placed edge-wise to the flow of air, which method ensures the maximum possible transfer to heat. Each chamber is fitted with three high speed centrifugal fan units.

Over 6 tons of coils are contained in each chamber which is rated at 360 kW's. Thermal uniformity is also furthered by dividing the rating of each chamber into three independent zones, each automatically controlled by an electric indicating controller. The furnace is designed for an operating temperature up to 650° C.

The rating of 360 kW's mentioned above, is reducible by means of Star/Delta switching during "soaking" periods, when an overall uniformity at the predetermined annealing temperature has been attained throughout the charge. This is an important factor in effecting considerable economy in operation.

The Institute of British Foundrymen

The Annual Conference held at Buxton

A very large number of members and visitors were present at this the 47th Annual Conference at which the facilities provided for technical discussions were comparable with previous Conferences, in consequence very lively and profitable sessions were held, in addition many functions were arranged which made the meeting a memorable one. Here it is only possible to summarise the early proceedings.

THE Annual General Meeting of the Institute of Foundrymen, held at Buxton, June 7-10 1950, maintained in many respects the high standards set by previous post-war annual meetings; the weather for the opening was what foundrymen had grown to expect, indeed a heat wave was experienced, and Buxton provided a setting that few districts can improve upon. An attendance of between 500 and 600 members and visitors enjoyed to the full a comprehensive programme prepared for them and the organising committee are to be congratulated on the success of their efforts.

The initial meeting was held in the ballroom of the Spa Hotel, and opened before a good attendance with the President, Mr. N. P. Newman, J.P., in the Chair, who extended a cordial welcome to overseas visitors, many of whom he named, and to members. The annual report for the year ended April 30th, 1950, which records a period of healthy activity and at the highest level commensurate with the financial resources available, was taken as read. The balance sheet for that year was also presented and, although the credit balance is not unsatisfactory, the Council has felt increasing concern regarding certain administrative and other expenditure, which shows a continuing tendency to rise, and deemed it advisable to recommend the adoption of a special resolution concerning an increase of subscription rates. This was moved and seconded at the meeting and was carried on a vote being taken. Thus, subject to the approval of the Privy Council, the rates of annual subscriptions commencing with January 1st, 1951, will be as follows: member, £3 13s. 6d.; associate member, £2 12s. 6d.; associate 21 years of age or over, 10s. under 21 years of age, 5s.; subscribing firm, 10s.

Medal Awards

Oliver Stubbs Gold Medal: Mr. J. Arnott, F.R.I.C., F.I.M. *E. J. Fox Medal:* Mr. S. R. Russell. *British Foundry Medal:* Mr. A. R. Martin, B.Sc., A.R.S.M. *Meritorious Services Medals:* Mr. T. R. Walker, M.A. and Mr. A. S. Wall.

Honorary Members

The distinction of Honorary Member was conferred upon the following members: JOHN CAMERON, Snr, J.P., in recognition of his services to the Institute and to the foundry industry; V. C. FAULKNER, in recognition of the contribution which he has made to the progress of the Institute and of the foundry industry; J. Hogg, in recognition of his outstanding administrative services to the Institute for many years and of the help and encouragement he has given to numerous foundrymen throughout the country.

Election of Officers

President: J. J. Sheehan, B.Sc., A.R.C.Sc.I., F.I.M.
Senior Vice-President: C. Gresty.

Junior Vice-President: C. H. Dadswell, Ph.D., B.Sc.

Elected to Council: Dr. A. B. Everest, R. L. Handley, Barrington Hooper, C.B.E., A. E. Peace, and R.C. Shepherd.

Induction of President

Mr. N. P. Newman, who inducted Mr. Sheehan into the Chair, referred to the Council's nomination for the Presidency of the Institute for the year 1950-51 as one of the most popular and highly esteemed figures in the Foundry industry. He had worked hard for the Institute having served on its Council and Technical Committees, and also on the Council of the British Cast Iron Research Association. He has presented many technical papers at the annual conferences and the Branch meetings of the Institute, and his efforts on behalf of the Institute, have been recognised by the award of the Oliver Stubbs Gold Medal. The new President suitably replied.

Presidential Address

Mr. Sheehan based his Address on what he referred to as a way of life in which it was pleasing to note the emphasis on work and play, with which, all their variations according to the different individual characteristics, constituted the fuller life. There are a lot of people who seem to have the impression that life only commences when the day's work is over, who regard working time as lost time. But it is largely during working time that character is formed, when sound friendships are fostered and appreciation of others can be more fully appreciated. At work the associations are much more varied and it is probable that, in the majority of cases, these have the greater influence on the complete life of the individual, and the foundrymen in particular, than the various interests of individuals that are completely separated from the environs of their work. Mr. Sheehan emphasised the advantages of active work in the Institute as a means of increasing personal contacts with those of similar interests, anxious to meet, converse, exchange knowledge, likely to lead to good fellowship and firm friendships. The Address was typical of the President and an inspiration to the members present.

Edward Williams' Lecture

The above lecture was given by Sir Andrew McCance, LL.D., D.Sc., F.R.S., whose subject, "Gases in Steel," was a particularly appropriate one before a meeting of foundrymen whose products are affected in varying ways by gas inclusions and consequently have considerable influence on soundness, which is a primary requisite in the production of castings. Sir Andrew dealt with hydrogen, nitrogen and oxygen as the main gaseous constituents of steel and reviewed the work on the subject by many leading chemists and metallurgists who have brought to light many interesting facts. In recent years, particularly, much experimental and co-ordinating work has been carried out and Sir Andrew gave a clear perspective of this once totally obscure field.

Hydrogen is absorbed by steel and assumes alloy characteristics. Contents as low as 0.0001% begin to have metallurgical significance. It is, therefore, important to know something about it and to devise dependable procedures for its accurate estimation. It was pointed out that, in general, the hydrogen content of molten or newly cast steel is appreciably higher than that of stored steel, but does not diminish appreciably after about a month when, however, the content can be as much as 0.0003% for a carbon steel or 0.001% for a chromium steel.

It is in its combination as nitrides that nitrogen calls for primary consideration in steels, although uncombined nitrogen sometimes exists, or a small proportion may be present in the form of cyanides, practically all the nitrogen present in iron and steel exists in the form of nitrides of the various constituents, usually with iron nitride and manganese nitride predominating.

Gaseous oxygen combines to form oxides directly with most elements, particularly at elevated temperatures, and indirectly with all elements, excepting the inert gases. Thus, since it combines with practically all the elements in iron and steel, usually to the disadvantage of the final composition, steps must be taken either to prevent its inclusion in the furnace charge or to reduce it in the molten steel.

It is impossible even to attempt to summarise the lengthy lecture delivered by Sir Andrew but the lucid manner in which he clarified a complex subject earned for him the attention of all present and in its complete form the lecture will provide a valuable source of reference for many years to come.

Technical Sessions

Some 13 papers and two technical reports were presented at this conference, they included "Some Properties and Applications of Spheroidal Graphite Cast Iron," by Dr. A. B. Everest; "Some Properties of Cast Iron at Sub-Atmospheric Temperatures," by C. N. J. Gilbert; "Experiences in the Application of Spectrographic Analyses in the Grey Iron Foundry," by E. J. Ronnie and M. M. Hallett, M.Sc.; "Non-Ferrous Investment Casting," by Hiram Brown, B.S.(Met.) (Exchange paper from the American Foundrymen's Society); "Sand-Cast Bars for Copper-Base Alloys," by Dr. O. R. J. Lee and B. W. Peck, B.Met.; "Some Experiments in Sand Casting of Conductivity Copper," by W. H. Glaisher; Report of Sub-Committee T.S.23 on "Repair and Reclamation of Grey Iron Castings by Welding and Allied Methods"; Report of Sub-Committee T.S.26 on "Repair and Reclamation of Non-Ferrous Castings"; "The Fluidity of Steel," by J. E. Worthington, B.Sc.; "The Effect of Liquid Metal Properties on the Casting Fluidity of Alloys," by Dr. V. Kondie; "The Deformation Characteristics of Five Grey Cast Irons at 400°C. and 500°C.," by C. R. Tottle, M.Met.; "Review of South African Foundry Industry," by H. G. Goyns; "The Rationalisation of Sand Preparation," by J. F. Goffart (Exchange paper from L'Association Technique de Fonderie de Belgique); "Testing the Metal or Testing the Castings"—Some Notes on the new Swedish Specifications, by E. Lissell, M.Sc.; "Similarities between Steels and Aluminium Bronze," by H. Laplanche (Exchange paper from L'Association Technique de Fonderie).

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INSTRUMENTS AND MATERIALS

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Design and Construction of Equipment for a Small Stress-Rupture Creep Laboratory

By F. C. Child, M.Sc., A.I.M.

Johnson, Matthey & Co., Ltd.

An account is given of experience in the development of a creep-testing laboratory for life-to-rupture tests on materials at 900° C. using miniature creep-testing machines, and details are given of the equipment finally installed. Although designed for high-temperature testing, the apparatus has given satisfactory performance, with only minor alterations, at temperatures as low as 400° C. Temperature control of $\pm 1^\circ$ C. at 900° C. can be achieved over periods of up to 1,000 hours, with a temperature gradient of less than 1° C.

THE creep-testing laboratory to be described was installed primarily for the investigation of cobalt-base alloys for operation at high temperatures in such plant as gas turbines. It was anticipated that large numbers of alloys would need to be examined and it was considered that life-to-rupture tests would provide a suitable basis for selecting promising alloys for future tests. Furthermore, since it was intended to use some of the rarer metals in many of the alloys, it was desirable to use as small test specimens as possible. For these reasons it was decided to employ Denison miniature creep-testing machines for the tests, as these take screwed specimens having a diameter of 0.1785 in. over a gauge length of 1 in.

I. Initial Installation

Initially, four machines were equipped with platinum-wound furnaces of the dimensions shown in Fig. 1.

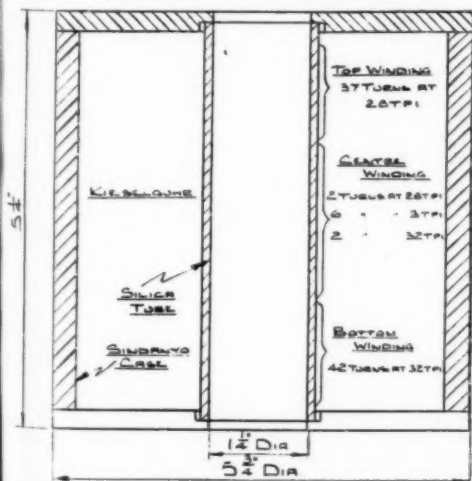


Fig. 1.—Dimensions of original furnace.

These dimensions were not considered to be ideal for the temperature at which the furnaces were required to run, but were determined by the design of the creep-testing machine and the furnace clamps. The furnace heaters were wound on silica tubes with 10% rhodium-platinum wire, 0.01 in. diameter, the windings being coated with alumina cement. The elements were mounted in Sindanyo cases and lagged with kieselguhr. After numerous trials the winding shown in Fig. 1 was adopted as being the most suitable, the requisite temperature being easily attained with a reasonably uniform temperature over the specimen gauge length.

The power control circuit is shown in Fig. 2. The three furnace windings connected in series were supplied through a small Variac transformer. The resistance R , in series with the furnace winding and shorted by the temperature controller, provided the means of temperature control, while the lamp shunts which could be switched across the top or bottom windings, together with the shunt resistance across the centre winding, provided for control of the temperature gradient. For temperature control, N.P.L. Prosser-type resistance thermometer controllers were constructed.

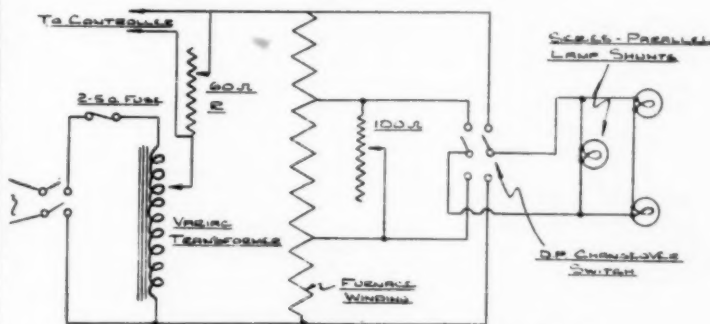


Fig. 2.—Original furnace power control circuit.

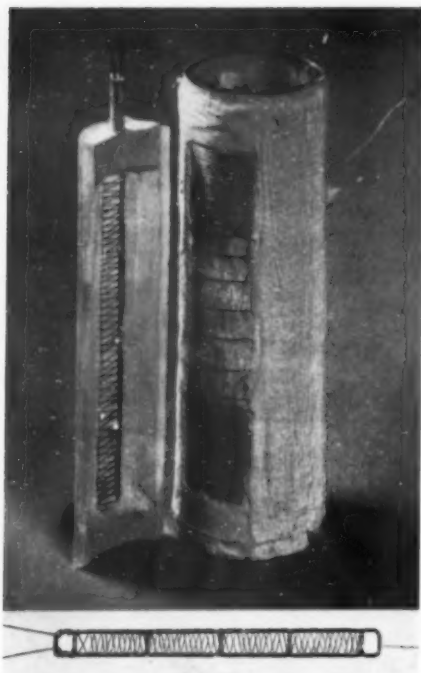


Fig. 3.—Furnace tube and resistance thermometer.

An initial attempt to control the temperature of all four furnaces by means of one controller operating on a fifth master furnace, all five furnaces being supplied in parallel from the same bus-bars with the same high power/low power ratio, was not entirely successful, and at a comparatively early date each furnace was equipped with its own controller. At the same time, modifications were made to the circuit of the controller to overcome certain defects (due mainly to sticking of relays), which the experience already gained had shown to be present in the original Prosser circuit. Additionally, new valve types had to be found to replace three of those originally recommended which had become obsolete.

In all these preliminary tests, the platinum resistance thermometer was placed with the specimen in the furnace tube. Owing to the limited space available, and the possibility of damage, it was later decided to situate the thermometer on the outside of the tube adjacent to the winding. The construction of the thermometer and the method of attaching it to the furnace are shown in Fig. 3.

The location of the thermometer in this position is open to the criticism that the temperature of the furnace winding and not the specimen temperature is being controlled, and hence there will be no compensation for variable heat losses by conduction along the shackles (probably the major form of heat loss). On the other hand, provided losses from the furnace are sensibly uniform at all times, control of the furnace winding temperature should result in better control of the specimen temperature since there can be little or no lead, or lag, between winding and thermometer temperatures; and, moreover, any temperature fluctuations will be smaller in amplitude inside the tube than outside owing to the damping effects of the walls of the furnace tube.

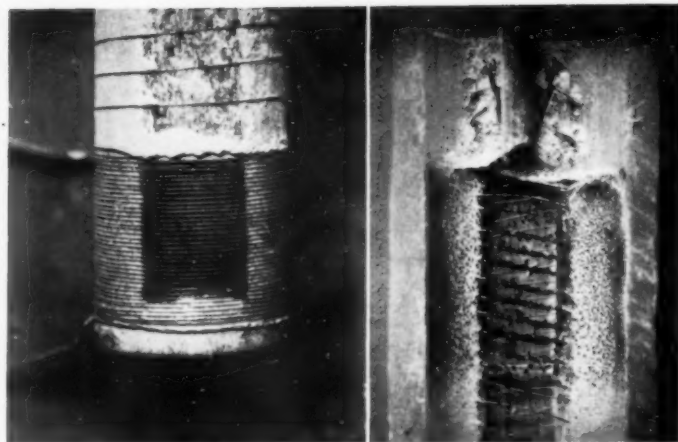


Fig. 4.—Volatilised platinum filling spaces between turns of the winding (left) and adhering to the resistance thermometer winding (right).

Variations in heat loss along the shackles should not be great unless the furnaces are placed in draughty situations, or unless the ambient temperature suffers gross variations. Since the room temperature had already been thermostatically controlled, and every care had been taken to ensure freedom from draughts, it was anticipated that the degree of temperature control would be as good as, if not better than, that obtained from an internal thermometer.

Specimen temperatures were measured in the usual way by means of three platinum/platinum-rhodium thermocouples attached to the top, centre and bottom of the specimen.

The equipment thus finally consisted of four creep-testing machines, each complete with furnace, furnace control panel and temperature controller. The furnaces and resistance thermometers for each machine were constructed as shown in Fig. 3; furnace control panels were to the circuit given in Fig. 2; and temperature controllers were laboratory modifications of the Prosser controller.

II. Defects in the Initial Installation

After the installation had been functioning for some time, and particularly during the period of heavy voltage fluctuations experienced during the winter of 1946-7, a number of defects in the equipment became apparent. The following section outlines the effects of these faults which were principally associated with the furnaces and their power control, and at the same time indicates the possible means of overcoming them. In most cases these means were successfully adopted in the final installation described in Section III.

(a) *Furnaces.*—It was noted at an early date that the average furnace life was considerably lower than was usual for a platinum wound furnace running at 900° C. and that difficulty was frequently found in attaining uniformity of temperature along the specimen, both initially and throughout the duration of a test. Both these troubles appeared to have their origin in the short furnace length. Reference to Fig. 3 will show that the furnace winding was continued practically to the end of the tube and was very heavily bunched at the ends, to overcome the high heat losses. This, of course, resulted in the end windings running at a temperature

considerably in excess of 900°C., a fact which was confirmed by examination of burned-out furnaces which, in most cases, showed considerable attack of the silica tube adjacent to the break in the wire, and very heavy volatilisation of the platinum (see Fig. 4). By filling the spaces between the turns with alumina cement but not covering the bare winding adjacent to the thermometer, volatilisation was reduced. One type of furnace failure, that due to bridging of the windings by platinum crystals, was thus overcome.

The second effect of the short furnace length was to make the gradient rather critically dependent on the position of the specimen, and it was found that small deviations of the specimen from the optimum position resulted in quite large changes in the temperature gradient, and as a consequence changes in gradient occurred during test due to elongation of the specimen. Despite the shunts provided to control the gradient, it was found that the winding had to be made with undesirable accuracy in order to produce a reasonably uniform temperature even with the specimen in the most advantageous position, and furnaces were quite frequently rejected as being incapable of producing a reasonable gradient—i.e., 2–3°C. over the 1 in. gauge length.

One further defect is worthy of mention at this juncture; a quite high electrical leakage occurred at the working temperature between the furnace winding and the resistance thermometer, and voltages of between 30 and 40 frequently existed between the thermometer leads and earth, with approximately 200 volts across the furnace winding. The construction of the furnaces wholly from alumina, in an attempt to improve furnace life, increased this leakage voltage to the order of 100–120 volts, due presumably to alumina having a higher conductivity at 900°C. than silica. The full effects of this leakage between winding and thermometer on the temperature control obtained will be dealt with in Section II c.

The solution to the difficulties so far noted appeared to lie principally in the construction of a longer furnace embodying the following features:—

- (i) A uniformly wound centre section long enough to cover the specimen and screwed-end grips, and with a sufficient allowance for extension of the specimen.
- (ii) End windings of sufficient power input to counteract heat losses via the shackles.
- (iii) A sufficient amount of lagging at each end to reduce the end losses to a minimum.
- (iv) A thermometer frame of length and strength adequate to allow its ends to be anchored at a distance from the winding, and to span the winding without intermediate support. This should reduce electrical leakage between winding and thermometer, partly by increasing the length of the electrical path, and partly by situating that path in the cooler parts of the tube where the resistivity of the refractory is higher.
- (v) The provision of earthed shields at the ends of the thermometer as a further safeguard against leakage currents from the winding.
- (vi) The construction of the furnace tube, etc., from alumina to improve furnace life.

After due consideration of these and certain other factors appertaining to gradient control (see section II b), a new type of furnace was constructed which has

proved to be free from all the faults mentioned (see Section III d).

(b) *Furnace power control.*—No major defects were found in the power control of the furnaces, but, as has already been mentioned, the lamp shunts across top or bottom did not prove capable of providing a really fine equalisation of top and bottom temperatures. The centre shunt, too, was found to be usually of little value, due no doubt to the very small heating effect of the centre winding.

Additionally, the alteration of a shunt across any one winding of the three series windings inevitably altered the current ratios in all three windings, thus making gradient adjustment largely a matter of intelligent guesswork. It was thought that if the three windings were supplied in parallel this interaction would be less obvious, and moreover, if reasonably wide and fine adjustment of the power input to each section were provided, the difficulty of eliminating temperature gradients in the specimen would be obviated, whilst no great accuracy would be necessary in the winding of the furnaces.

Of the several methods possible of supplying the windings with the necessary comparatively low voltages, variable within wide limits, the use of variable transformers, although somewhat expensive, appeared to be the neatest and best from the point of view of power economy. Accordingly a control panel and furnace were constructed along these lines, and after minor modifications proved very satisfactory. This type of control unit was incorporated in the final installation and is fully described in Section III c.

(c) *Temperature controllers.*—The temperature control achieved on the furnaces, although usually just within specification limits, was obtained only with difficulty, but this was ascribed mainly to the defects in furnace design already noted rather than to the temperature controllers themselves.

In Section II a the existence of a high leakage voltage on the resistance thermometer from the furnace winding was mentioned. It will be obvious from a consideration of the circuit of the controller (see Fig. 10) that the presence of such a parasitic voltage should have an effect on the controller, but provided the voltage remains constant, within fairly fine limits, and no erratic phase changes take place, experience has shown that the controllers will continue to function reasonably well. If, however, the leakage voltage is subject to variations, as it must be since it is dependent on the voltage applied to the furnace windings, then the temperature control will be affected in sympathy with voltage variations on the windings. During the winter of 1946–7 when gross variations of mains voltage were encountered (180–250 volts), this fault in the furnace design caused large deviations in temperature. The elimination of this defect has, however, been dealt with in Section II a.

Only one trouble was encountered in the action of the controllers themselves, and this was due to faulty component design. The slide wire, or potentiometer, incorporated in the A.C. bridge circuit and controlling the temperature setting, has obviously to be as free as possible from all contact resistance variations. The potentiometers used in the controllers were of a commercial type employing Constantan or Eureka wire wound on a fibre former; the moving contacts were of a not very satisfactory nature and, unless periodically cleaned,

the variations in contact resistance were sufficient to make fine control of temperature impossible. In addition, the wire periodically became loose on the former due either to shrinkage of the former, or to stretching of the wire caused by the contact rolling on it. To overcome these troubles, a new potentiometer (see Section III e) incorporating robust non-tarnishable contacts and tarnish-resistant wire was constructed, and proved to be a satisfactory solution to the defects encountered in the previous model.

A single creep-testing unit incorporating all the modifications so far indicated in furnace, furnace power control and temperature controllers gave very satisfactory results over a period of several months. During this period it was decided to increase the number of testing units from four to eight, and to move the laboratory to a new situation. The design of the equipment for the new laboratory was based on that of the single prototype unit which had so far proved successful.

III. Final Installation

(a) *General.*—A block plan of the layout in the new laboratory is given in Fig. 5. The room temperature is thermostatically controlled by means of a refrigeration plant and auxiliary heaters at 65° F. in the winter and 75° F. in the summer.

The eight creep-testing machines are mounted on steel tables fitted with levelling screws, four machines on a single central table, and two on each of two tables against the side walls. The furnace control panels, temperature controllers, etc., are mounted in a single unit along one wall of the laboratory and flanked on either side by a constant voltage regulator and a small bench.

Previous experience had shown the necessity for assembling the apparatus on the unit principle and arranging for controllers, control panels, etc., to be removed for servicing with a minimum of time and trouble. Considerable care has been taken to attain this end, and in the present set-up a defective controller can be replaced in 5 or 6 minutes.

(b) *Central control panel.*—The electrical control gear is all mounted in the single unit shown in Fig. 6. The upper half of this is divided into eleven panels, four

down either side carrying the individual furnace control panels, the centre top carrying eight time-indicating clocks, the centre panel a 6-in. scale voltmeter and ammeter, while the lower centre panel gives access to the power distribution board.

The main power supply is from a Breco constant-voltage transformer capable of maintaining an output voltage of 240 volts $\pm 1\%$ for mains voltage fluctuations of 180–260 volts. The output from the regulator is taken to a nine-way distribution board inside the panel. A separate circuit is used to feed the control panel and temperature controller for each testing unit, whilst the ninth circuit supplies the time clocks, a remote-controlled thermocouple selection device and a point on the bench used for testing controllers, each of these points being individually fused.

The voltage-dropping resistances for the furnaces are mounted behind their respective furnace control panels and are accessible through the sides of the unit.

The lower half of the unit carries the eight electronic temperature controllers.

(c) *Furnace power control.*—Fig. 7 shows front and back views of a furnace control panel and Fig. 8 the wiring diagram. Three Variac transformers marked Top, Centre and Bottom, control the power input to the top, centre and bottom sections of the furnace windings, respectively. These three Variacs are supplied in parallel by the fourth Variac (Main), which therefore provides the means of adjusting the whole power to the furnace without alteration to the proportioning of power to the three zones. A resistance in the primary side of the Main Variac, shorted by the temperature controller, provides the means of temperature control. This resistance is adjusted so that only 10–20% of the power input is switched on and off for control purposes.

A three-pin socket and switch are provided to enable the voltage and current output of each Variac to be determined when necessary, while fuses protect each transformer from damage due to overload.

All connections to the rear of the furnace panel are made by plugs and sockets to facilitate the removal of the panel from the unit for servicing. A three-pin plug connects the power supply to the panel, a two-pin plug connects the panel to the voltage dropping resistance,

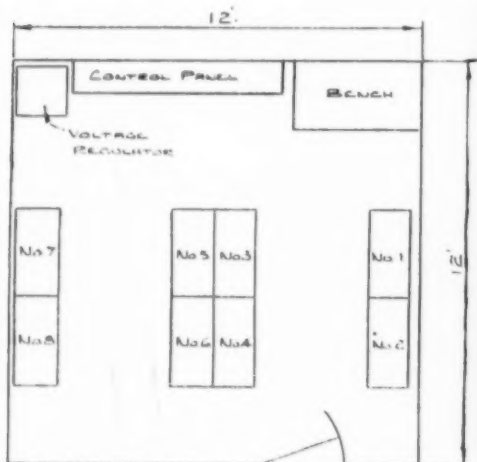


Fig. 5.—Block plan of creep laboratory.

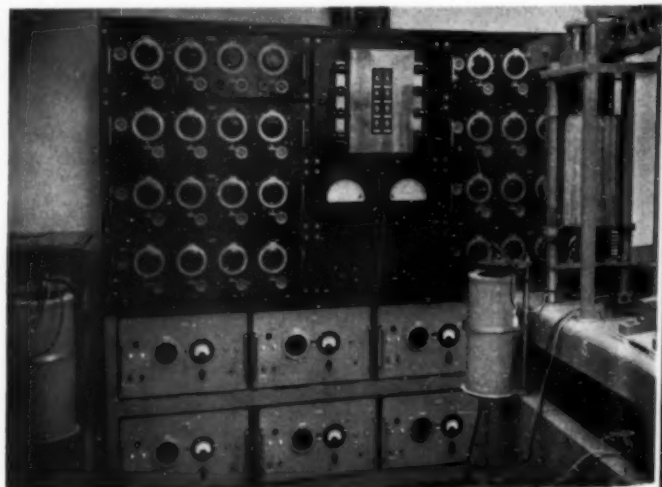


Fig. 6.—Central control panel.

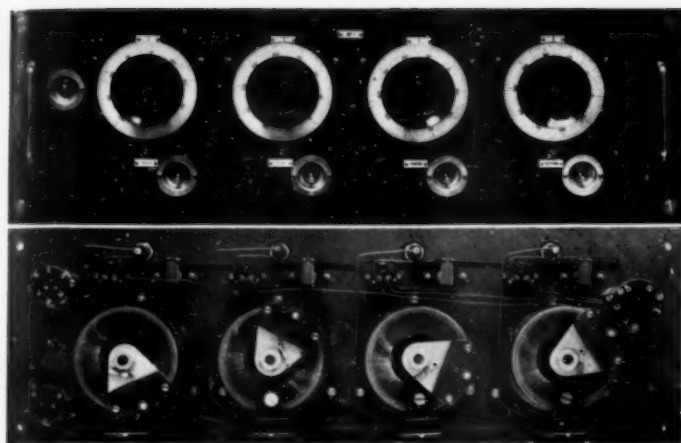


Fig. 7.—Front and rear view of furnace control panel.

whilst a six-pin plug (five points only in use) connects the five-core cable carrying the three live leads, the common neutral, and the earth from the furnace to the panels.

(d) *Furnaces*.—These are wound on tubes approximately $8\frac{1}{2}$ in. long \times $1\frac{1}{4}$ in. internal diameter fabricated from alumina cement. The winding of 10% rhodium-platinum wire, 0.01 in. diameter, is made uniformly at 32 turns/in., but is divided into four sections, two end sections each of 36 turns, and two centre windings of 48 turns. The division of the centre winding into two sections in parallel is necessary to avoid having high and low voltages side by side. Small alumina pads are provided on the tube at a distance of approximately $\frac{1}{2}$ in. from either end of the winding and of sufficient height to support the resistance thermometer frame $\frac{1}{2}$ in. from the winding.

The resistance thermometer consists of 4 ft. of 0.008 in. diameter thermopure platinum wire wound non-inductively on an alumina cement frame $7\frac{1}{2}$ in. long \times $\frac{1}{2}$ in. wide \times $\frac{1}{2}$ in. thick. Slots are made in the frame over the winding length to expose the greatest possible area of wire to direct radiation from the furnace winding. The resistance thermometer winding is wound to cover only the two centre windings—i.e., that portion of the

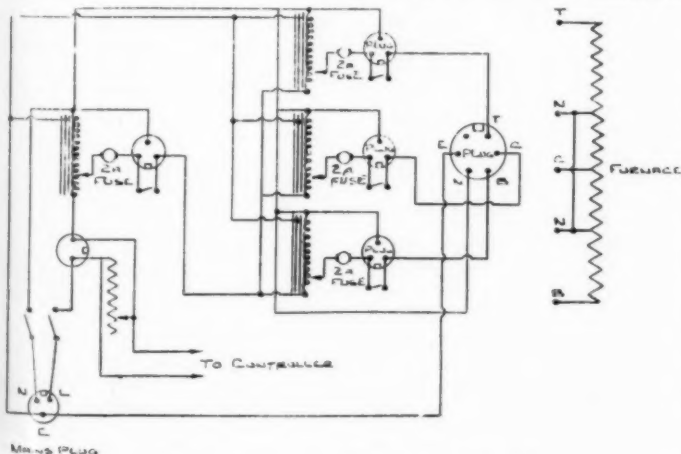


Fig. 8.—Furnace power control circuit.

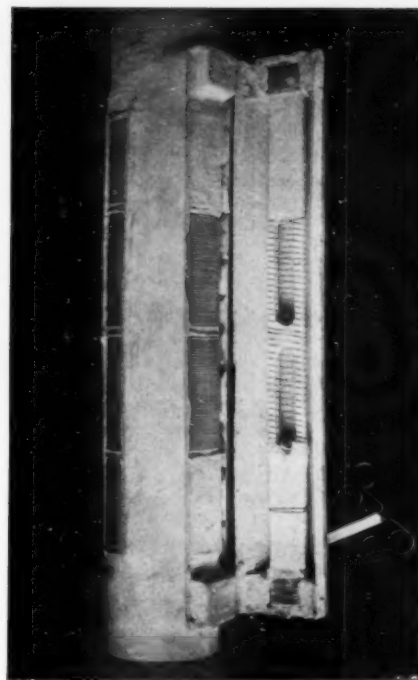


Fig. 9.—Furnace tube and resistance thermometer.

furnace in which the specimen is situated. A thin layer of alumina cement down the edges of the thermometer frame serves to secure the winding in place. Thermopure platinum leads 0.02 in. diameter are attached to the ends of the winding and compensating leads of the same material are run to the junction of the winding and lead. A spiral of platinum wire at each end of the thermometer frame acts as an earth shield.

The furnace winding is covered with alumina cement carefully worked between the windings, a necessity if volatilisation of the winding is to be reduced to a minimum, except for the centre portion immediately facing the thermometer. The winding over this portion is filled, but not covered, with cement to allow of maximum radiation. Finally, an alumina cover to the resistance thermometer is cemented in place.

Fig. 9 shows the furnace unit with the thermometer and its cover tube opened from the winding; a strip of cement has also been omitted from the furnace winding.

The completed unit is mounted in a polished aluminium case approximately 9 in. long \times $6\frac{1}{4}$ in. diameter, with recessed Sindanyo end plates to position the tube. Kieselguhr is used as the insulating packing. All furnace power and earthing connections are brought to a terminal board on the side of the furnace. The thermometer and compensating leads are brought straight through the furnace wall in quadruple bore alumina tubing and soldered direct to the copper leads.

The completed furnaces maintain the specimens at 900° C. with a power consumption of approximately 375 watts.

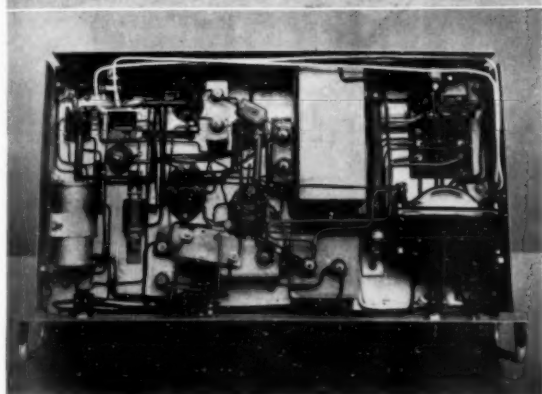
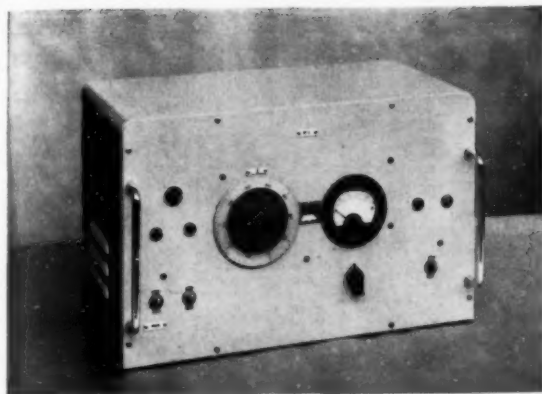


Fig. 11.—Furnace temperature controller.

action tends to obviate troubles from dust, etc., whilst the direct rotation of the drum obviates all "backlash."

Although other designs of potentiometer are possible with three, or only two moving contacts, positive motion of the contact in step with the wire is difficult, if not impossible, and in the absence of this positive motion there is always the probability of the contact jumping off the wire unless it is in the form of a rolling wheel. This, however, is undesirable since this type of contact is not self-cleaning.

(f) *Creep machines.*—The only alteration made to the creep units as supplied was the removal of the rather cumbersome furnace clamping arrangement. The furnaces are now attached to the machines by two small

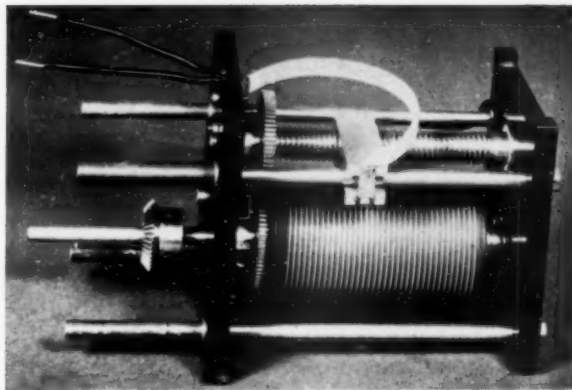


Fig. 12.—Potentiometer.

lugs which bolt on to two loose fitting collars on one pillar, thus allowing the furnace to swivel on the pillar.

Microswitches have been mounted under the weight-holder of each machine and are connected to the time-clocks on the main panel. Precautions have been taken to prevent damage to the microswitches by the falling weights.

Fig. 13 shows a single unit with a test in progress.

(g) *Temperature measurement.*—The temperature of the specimen is determined by three platinum/platinum-rhodium thermocouples secured to the top, centre and bottom of the test piece and insulated by twin bore alumina tubing. Originally asbestos paper pads were used to protect the thermocouples from direct radiation, but experience showed that contamination was occurring and this practice has now been abandoned.

Ice flasks attached to each machine contain the cold junctions of the thermocouples, and flex leads are taken from the cold junctions to a 25 point post-office-type selector switch. This switch is remote-controlled from the potentiometer (60–70 yds. away in the adjoining laboratory) with which all temperature measurements are made. An indicator board mounted near the potentiometer shows which thermocouple is connected to the instrument.

(h) *Connections between the various units.*—All inter-unit wiring is carried on wooden bearers on the walls, or overhead to the centre machines. A single five-core cable connects each furnace panel to a plug fixed on a small panel on the stand of the machine, while two twin single-core cables connect the A.C. bridge circuit in each controller to solder tags on the same panel. Short leads connect the furnace power terminals to the plug and the resistance thermometer and compensating leads to the solder tags.

The apparatus described was installed primarily for creep tests at temperatures of the order of 900° C., but it has been found to be capable of giving almost equally satisfactory results at 400° C. with only two minor alterations. The voltage dropping resistance requires to be increased with decreasing temperature, and the resistance B_3 (Fig. 10) in the A.C. bridge of the controller has to be reduced to a value approximately equal to that of the resistance thermometer at the required furnace temperature. This latter adjustment is necessary since the temperature range covered by the potentiometer is of the order of 200° C.

Table I gives the temperature readings taken over a

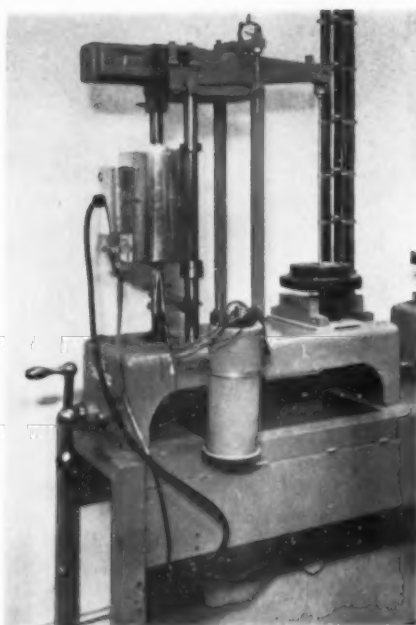


Fig. 13.—
Creep
unit with
test in
progress.

period of several days in the course of a test and shows that gradients in the specimen can be maintained at

TABLE I.—RECORD OF TEMPERATURES ON AN ACTUAL TEST

Date	Time	Hours of Test	Temperatures			Adjustments
			Top	Centre	Bottom	
5.1.49	10-20 a.m.	0	900-2	899-9	899-3	Bottom up 0-02 amp.
6.1.49	5-0 p.m.	7	899-7	899-8	899-5	
7.1.49	9-30 a.m.	23	899-5	899-7	899-5	
7.1.49	9-30 a.m.	47	899-8	899-8	899-5	
11.1.49	9-30 a.m.	143	899-7	899-5	899-4	Bottom up 0-02 amp.
12.1.49	9-30 a.m.	167	900-1	900-0	899-5	
13.1.49	9-30 a.m.	191	900-2	900-2	899-8	
20.1.49	9-30 a.m.	329	900-0	900-0	899-6	
21.1.49	9-30 a.m.	383	899-9	900-0	899-8	
24.1.49	9-30 a.m.	456	900-2	900-2	900-1	
25.1.49	9-30 a.m.	479	900-5	900-0	898-9	
2.2.49	4-0 p.m.	480	899-9	899-8	899-2	
2.2.49	9-30 a.m.	671	899-9	900-5	900-2	
3.2.49	9-30 a.m.	695	900-1	900-3	900-2	
4.2.49	9-30 a.m.	719	900-2	900-5	900-3	
7.2.49	9-30 a.m.	793	900-1	900-2	900-2	
22.2.49	9-30 a.m.	1151	900-0	900-2	899-8	
23.2.49	9-30 a.m.	1175	899-9	899-9	899-5	
24.2.49	9-30 a.m.	1199	899-8	899-8	899-6	

better than 1°C. , whilst temperatures can be controlled to $\pm 1^{\circ}\text{C.}$ at 900°C. over periods of up to 1,000 hours. Since the limits of accuracy of the thermocouples in such circumstances are probably of the same order, attempts at closer control than this would be pointless.

Acknowledgments

The author's thanks are due to Messrs. Johnson, Matthey & Co., Ltd., for permission to publish this paper. Acknowledgments are also due to his colleagues for their advice and assistance.

Miscellaneous Microchemical Devices (XXVI) Sulphide Precipitation by the Pressure Method

By J. T. STOCK and P. HEATH

IN the performance of qualitative analysis on the ordinary scale, treatment with hydrogen sulphide is effected either by allowing the gas to bubble through the test solution, or by the "pressure" method. In the latter method, the solution is placed in a small flask carrying a well-fitting stopper and a lead-in tube. Having displaced the air in the flask by loosening the stopper and opening the generator stopcock, the stopper is thrust firmly home and the flask is well shaken at intervals until the solution becomes saturated with gas. A considerable saving of hydrogen sulphide and lessened contamination of the laboratory atmosphere thereby result. However, the experiments of Belcher¹ and his co-workers on the precipitation of cadmium sulphide have shown that the saturating efficiency of the "pressure" method is by no means as high as is usually assumed, the bubbling method being more satisfactory.

On the micro or semi-micro scale, the greater area-to-volume ratio of the sample renders easier the saturation with gas. However, since little hydrogen sulphide is used in any micro-procedure, the advantages of the "pressure" method referred to above scarcely apply. A point of very definite interest to teaching

laboratories is that an ordinary Kipp generator may be used, the high-pressure generators² desirable for use with the small-bore delivery tubes used in the "bubbling" method being quite unnecessary.

Precipitation is carried out in a 75 mm. \times 10 mm. test tube as shown in Fig. 1. The delivery attachment is a 70 mm. length of 5 mm. outside diameter glass tubing carrying a short piece of rubber tubing fitting snugly in to the mouth of the test tube. At its lower end, the bore of the delivery tube is reduced to about 0.5 to 1 mm. After connecting to the generator, the attachment is loosened in the test tube and the generator stop-cock is opened slowly until about 10 to 15 bubbles of gas have passed through the gas-washing bottle. The attachment is then thrust firmly home in the mouth of the test tube and, with the stop-cock open, the test tube is gently shaken for about a minute, avoiding splashing as much as possible. Having stopped the gas supply, the test tube is detached, the contents rapidly centrifuged, and the treatment repeated to test for completeness of precipitation. A few drops of distilled water are then run on to the surface

of the solution; further precipitation (e.g., of cadmium sulphide) at the liquid junction indicates that the initial acidity was too high.

With reasonable care, little contamination of the delivery attachment occurs, and thorough rinsing immediately after use is all that is usually necessary. As a precaution, batches of attachments may be boiled-out periodically in dilute nitric acid, and, when the formation of an intractable deposit does occur replacement entails but little time or expense.



Fig. 1. Adaptor for Sulphide Precipitation.

¹ Belcher, B., *Jour. and Proc. Roy. Inst. Chem.*, 1949, 101.

² Liddstone, A. G., Wilson, C. L. and Wilson, D. W., *Metallurgia*, 1947, 35, 171; Stock, J. T. and Heath, P. *ibid.*, 1950, 41, 171.

Apparatus for the Measurement of Creep Under Fluctuating Stress

By W. R. Tyldesley, M.Sc.

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An apparatus is described for the measurement of creep in wire specimens under a fluctuating stress, a subject which has received comparatively little attention. The stress system comprises a steady load on which is superimposed a smaller load fluctuating at the rate of 100 cycles per second. The equipment has been applied to the study of the creep of lead at room temperature.

MEASUREMENTS of creep have been chiefly carried out under constant tensile load or constant stress, whereas creep under fluctuating stress has received little attention. In his early work, Archbutt¹ carried out tests with specimens subjected to vibration and found an increase in the creep rate. Recently, work in this field has been reported by Greenwood and Cole² and Greenwood.³ In this work the fluctuating stress component was introduced by the rotation of an off-balance flywheel driven by an electric motor mounted on the frame from which the dead-loaded test pieces were suspended. The vibration from the asymmetric load was transmitted to the specimens by the motion of the whole machine frame. Measurement of the amplitude of the vibration was made, but no attempt was made to measure the stress variation which, in view of the amplitude measurements, would seem to be of a complex nature.

The present apparatus was designed with the intention of carrying out creep tests in which the load included a fluctuating component, the magnitude and regularity of which would be under control. The fluctuating load is provided by the attraction of a soft iron draw mass by an A.C. solenoid. Using the mains supply of frequency 50 cycles per second, a fluctuating load of frequency 100 cycles per second is obtained. The fluctuating component is applied together with a constant dead load and is small compared with the constant load.

The range of the machine is somewhat restricted, it being designed for measurement of approximately 0.2 in. creep on an 8 in. wire specimen. There is, however, no reason why the principle should not be applied to the measurement of greater degrees of creep.

Apparatus

The loading unit of the machine is shown in Fig. 1. A hollow soft-iron core *A* is fitted with ebonite flanges *D* $4\frac{1}{2}$ in. in diameter, the $\frac{3}{4}$ in. diameter core being slightly stepped in order to retain the flanges tightly. Through the hollow core passes, with 0.01 in. clearance, a $\frac{1}{4}$ in. brass rod *E* on which a soft-iron cylinder *B*, 2 in. long by $\frac{3}{4}$ in. diameter, is fixed by means of two set screws. The rod can thus slide through the core of the magnet until the end of the cylinder is in direct contact with the end of the core, the contacting faces of the core and cylinder being machined flat. The coil is wound with 2,000 turns of 18-gauge double silk covered copper wire *C*

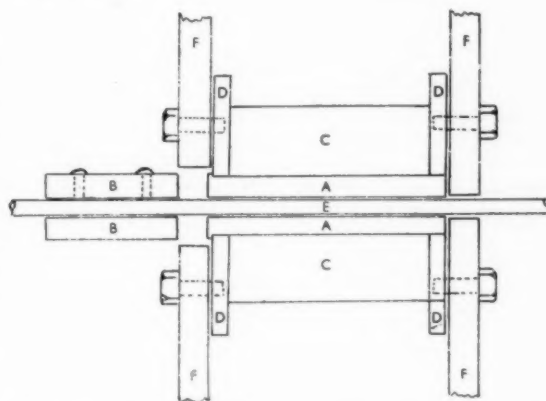


Fig. 1.—Loading unit of fluctuating-stress creep-testing equipment.

and the ebonite flanges are drilled and tapped to take $\frac{3}{8}$ in. Whitworth bolts for fixing to the frame of the machine *F*.

The ends of the brass rod are threaded with $\frac{1}{4}$ in. Whitworth threads, one end being screwed into a simple screw down brass grip, and the other end into the extensometer unit. In the initial experiments, the latter consisted of a simple lever by which the motion of the brass rod *E* (Fig. 1) was transmitted to a dial gauge graduated in thousandths of an inch. It is now replaced by the system to be described below.

The back grip of the machine is bolted to a weigh bar of spring steel, $\frac{1}{8}$ in. by $1\frac{1}{2}$ in. by 9 in. long, which is bolted rigidly to the frame of the machine. The spindle of a dial gauge reading to 0.0001 in. is maintained in contact with the flat back surface of the weigh bar by the spring action of the gauge. The deflection of the weigh bar can thus be directly measured.

Cemented to the weigh bar are electrical resistance strain gauges which may be connected through a simple amplifying system to an oscillograph for the measurement of cyclic deflections in the weigh bar. Alternatively, the gauges may be connected to a Kelvin double bridge for the measurement of static deflections in the weigh bar.

The loading and extensometer unit and the back-grip unit are bolted down to a base plate 3 ft. by 8 in., the supporting framework being of $\frac{1}{2}$ in. by $1\frac{1}{2}$ in. aluminium bar in the case of the loading unit, and of 1 in. channel

Archbutt, *Trans. Farr. Soc.*, 17, 1921, p.22.
Greenwood and Cole, *Metallurgia*, 37, 1948, p.285.
Greenwood, *Am. Soc. Testing Materials*, Preprint 29, 1949.

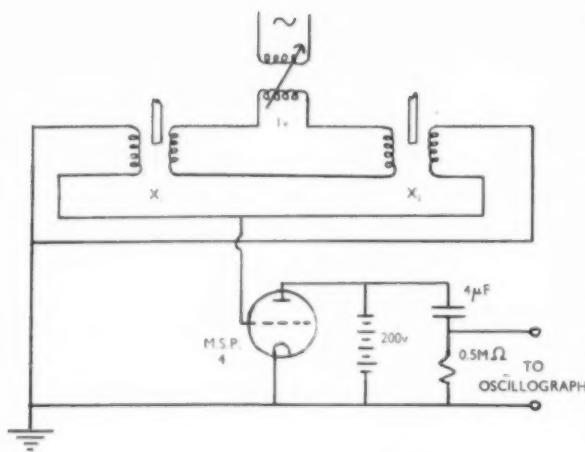


Fig. 2.—The electrical system.

section iron in the case of the extensometer and load measuring units. The brass rod is supported in the loading unit by a lubricated $\frac{1}{4}$ in. diameter tool steel rod held at right angles to it. This steel rod is adjustable in height by means of two bolts, through the heads of which the steel rod passes, held tightly to the base by means of two pairs of nuts.

The electrical leads to the coil are brought through slots in the ebonite flanges and are connected to terminals on the base plate of the machine.

Preliminary tests, with alternating current passing in the magnet, suggested that the extensometer system first used—a simple dial gauge arrangement—was unsatisfactory, as the vibration of the needle of the extensometer dial gauge was so great that no accurate readings could be obtained. A contacting micrometer head system was arranged, but again the vibration was far too great to allow for readings of any accuracy. In view of this problem of vibration, an electrical inductive extensometer has been built in which contacting parts are eliminated.

The electrical system is shown in Fig. 2. The two pairs of inductive coils X_1 , X_2 , consist each of a primary and a secondary winding of 210 turns each in six layers, the two units being identically wound on hollow brass tubes with ebonite end flanges. Into this tube can be moved, in each case, a length of $\frac{1}{4}$ in. diameter soft steel rod. The output from the secondaries of each pair are superimposed in opposition, the resulting combined output being amplified some twenty times by the simple single-

stage circuit shown. The output is fed to the amplifier of a Furzehill type D 1684D/2 oscillograph. The voltage supply to the coils X_1 , X_2 is obtained from a Variac auto transformer.

One of the coil units X_1 is mounted on the extensometer unit of the machine, the steel rod being hinged to the extensometer arm. In this way the extension of the specimen results in a movement of the steel rod into the coil, the magnitude of the movement being doubled by the magnification due to the extensometer arm arrangement. The second coil unit X_2 is mounted on a separate small frame and the accompanying steel rod is mounted axially on the barrel of a micrometer head so that it may be moved into the hollow core of the coil, the magnitude of this movement being read on the micrometer head. The two coil units are shown in Fig. 3.

In Fig. 4 the extensometer is shown set up on the machine, and in Fig. 5 is shown the machine with its associated apparatus set up for use.

Technique

In creep testing it is essential that the load or the stress shall be sensibly constant over the whole range of creep. In the case of the present apparatus difficulty was anticipated in this matter of applying a fluctuating load of constant magnitude to an extending specimen. By the nature of the apparatus an extension of the specimen is accompanied by a closer approach of the iron draw mass to the magnet core. It was necessary, therefore, to ascertain whether the increase in load resulting from the inverse square law effect was of significance in the range of creep over which the machine might be used.

The weigh bar was first calibrated by dead loading, the deflection being directly measured by the dial gauge attached to the frame. A $\frac{1}{4}$ in. diameter steel rod was clamped between the grips of the machine, this rod being regarded as completely rigid for the loads under consideration. The air gap between the solenoid core and the draw mass was then set at 0.25 in., as being a reasonably likely value, and a direct magnetising current was passed through the solenoid to produce a deflection in the weigh bar corresponding to a load of 5 lb. The air gap was then varied by altering the position of the draw mass over a range giving gaps of from 0.1 to 0.4 in. This was repeated for magnetising currents corresponding to loads of 2, 3, 4 and 6 lb.

It was found that within the limits of loads from 2 to 4 lb. varying the air gaps from 0.3 to 0.1 in. had no appreciable effect on the magnitude of the load. Thus, provided the fluctuating component of the load has a maximum value within the indicated limit, and provided

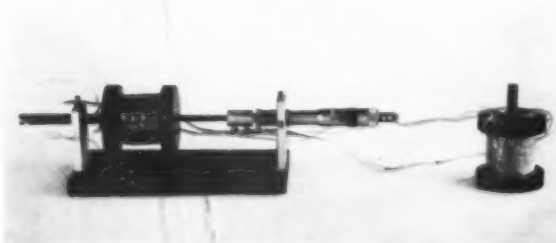


Fig. 3

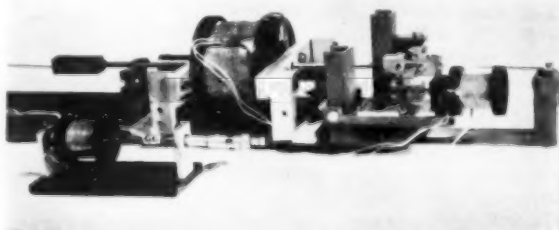


Fig. 4

Fig. 3.—The coil units. Fig. 4.—General view of the loading end of the equipment, showing the vibration unit and extensometer.

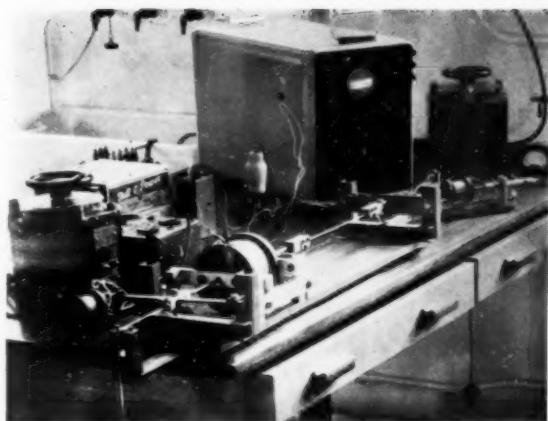


Fig. 5.—General view of the complete equipment.

the magnitude of creep is also within the limiting values, no error is introduced by the effect of the inverse square law of magnetic attraction and the fluctuating component of the load remains unchanged over the range of creep.

The form of the fluctuating load may be shown, by the variation in the resistance of the strain gauge cemented to the weigh bar, to approximate closely to sinusoidal. The value of the fluctuating load may be calculated from the calibration curves obtained using direct current in the solenoid. The mean value of the load component added by an alternating current is equal to that given by an equal direct current, the fluctuating component having a half amplitude of $\sqrt{2}$ -1 times the mean value. The calculated values for the mean and fluctuating components added by the electromagnetic field have been checked over a range of values by the use of the strain gauge assembly on the weigh bar, and have been shown to be of sufficient accuracy to enable the component to be measured in this way.

Two techniques have been investigated for the use of the extensometer. A direct reading method was first used, the basis of which is to balance the output from the two inductive coils by adjusting the micrometer head after the creep specimen has been set up, and immediately preceding the commencement of the test. An increase in the length of the specimen is then followed by movement of the steel core into the extensometer coil and hence by the unbalancing of the electrical circuit. The out-of-balance alternating voltage thus produced is directly measureable as a simple sine wave on the oscillograph screen. A simple calibration of the apparatus gives the relationship between extension in the specimen and size of image on the screen. It is found that, with the maximum amplification of the circuit used, a motion of 0.001 in. in the extensometer (of half that magnitude in the creep specimen) results in an increase of some 4 mm. in the amplitude of the measured sine wave.

This direct reading method has, however, been abandoned, as a considerable zero error is brought about by the electrical non-identity of the two pairs of inductive coils. Although extreme care was taken to ensure physical similarity, slight differences do, in fact, occur, of sufficient magnitude to render a perfect balance impossible. The zero reading appears, not as a straight line on the oscillograph, but as a much distorted sine

wave. This zero sine wave is of small amplitude but makes the zero reading uncertain.

The non-linearity of the method is also a serious objection. Although a linear relationship between movement in the specimen and amplitude of the measured wave does set in after some small extension, the first stages are of a more complicated nature. Another less fundamental, but none the less real, objection to the direct reading method is the limitation placed upon the range of the extensometer by the diameter of the oscillograph screen. When the amplitude of the measured wave approaches the diameter of the screen the only way of continuing the measurement is to operate the Y-shift, to bring the top of the wave to the centre line of the screen and to measure the increase in amplitude of the half wave. This is clearly unsatisfactory since it is impossible to place the image accurately so that the true half wave shall be measured, and since the sensitivity of the apparatus is reduced by one-half.

From these considerations it may be concluded that a zero method is more satisfactory. After setting up the test specimen the micrometer head on the second coil unit is fully extended and the steel core thus removed from the coil. The core is then slowly moved into the coil by means of the micrometer head until the sine wave on the screen is of minimum amplitude. The penetration of the core into the coil is continued until the observed trace is of a considerable magnitude—two or three screen diameters. The Y-shift is then operated to bring the top of the trace in line with the centre of the screen. The extensometer is now ready for use, the readings being taken on the micrometer head whilst the tip of the trace is maintained at the centre line of the screen.

Considerable experiment has shown that a simplification in the readings is brought about if the time base of the oscillograph is dispensed with. In this case the trace appears on the screen as a vertical line which is easily centred and the end of which is rather more clearly defined than the tip of the sine wave.

Applications

In Fig. 6 is illustrated a test in which a specimen of lead wire, of 0.08 in. diameter, was creeping under a load of 4 lb. and after 15 minutes had applied to it a fluctuating component of mean value 1 lb.

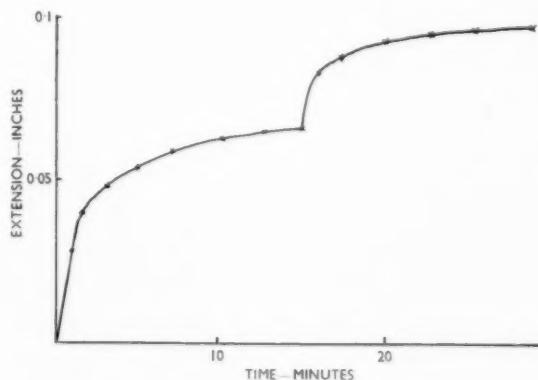


Fig. 6—Typical curve obtained with the apparatus.

This is typical of the type of test for which the apparatus is suitable. It is designed for low degrees of creep and, as has been indicated, the constancy of the electromagnetically added component becomes open to doubt for creep greater than 0.2 in. Within the indicated limits, however, the apparatus gives consistent and satisfactory results.

Acknowledgements

In conclusion the author would like to express his thanks to Professor F. C. Thompson in whose laboratories the work was carried out and to the Department of Scientific and Industrial Research (Ministry of Supply) for financial assistance.

Simple Apparatus for Micro-Conductometric Titration

By J. T. Stock

(Chemistry Dept., L.C.C. Norwood Technical College, London, S.E.27)

A simple mains-operated conductometric titration apparatus using micro-electrodes constructed from platinum wire is described. Samples of volume less than 1 ml. may be titrated with it. The relationship between electrode area and polarisation is discussed, and methods of stirring and other points of technique are briefly reviewed.

SINCE, as in other electrometric titration techniques, the electrical measurements are made only with a view to following of the course of a chemical reaction, absolute values of conductivity are not required in conductometric analysis. Accordingly, it is sufficient to ensure that the design of the electrodes is appropriate to the range of concentrations envisaged: a knowledge of the "cell constant" is not required. Similarly, unless there is some particular practical reason for operating under special conditions, the temperature, provided that it is constant, need not be fixed. Again, change in conductivity with frequency of the alternating current supply is immaterial if this frequency is constant. For this reason, alternating current drawn from the 50-cycle mains has on many occasions been suggested for conductometric titration, and it has been repeatedly pointed out that, if the voltage applied to the electrodes remains constant, then the current flowing through the solution is proportional to the conductivity of the solution. Thus the titration may be followed by placing an A.C. current meter in series with the electrodes and the A.C. supply, so that the sole manipulation during the titration is the operation of the burette.^{1,2} Usually, the "A.C. meter" is a D.C. instrument used in conjunction with a valve, crystal, metal-oxide, or mechanical rectifier.³ The scale readings are then usually arbitrary, but this is immaterial provided they are directly proportional to the flow of current through the solution.

Mains-operated Apparatus

The circuit of a simple apparatus of this type is shown in Fig. 1. Surplus radio material features largely in the construction of this titration unit, which is built into a vertical cabinet 7½ in. high × 7 in. wide × 4½ in. deep. Since teaching and demonstration purposes were envisaged, a Perspex back is fitted, and a sloping panel of the same material carries the appropriately labelled controls and a pilot light. Power from the A.C. mains, controlled by switch *S*, is fed to the primary of a small step-down transformer *T*, of the type used for operating

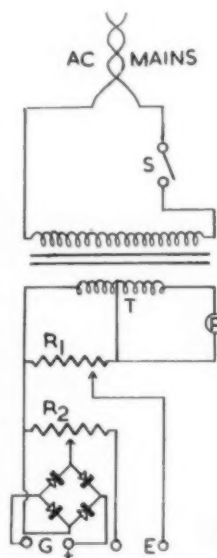


Fig. 1.—Circuit of titration unit.

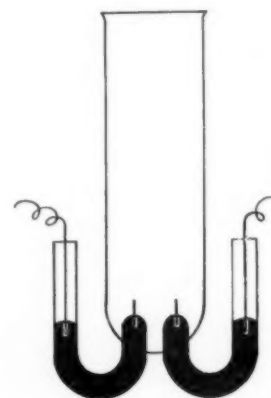


Fig. 2.—Callan and Horrabin cell.

electric bells. The output from one half of the secondary is connected to pilot lamp *P*, which serves as a reminder to switch off after use. Potentiometer *R*₁, of resistance about 15 ohms, is connected across the other half of the secondary and allows any A.C. voltage up to about 2.5 to be applied to the electrode terminals *E*. This control is fitted with a graduated paper scale to enable a particular voltage setting to be duplicated. If a radio test meter or other A.C. voltmeter is available, calibration (even roughly) in tenths or quarters of a volt may be advantageously carried out.

Sensitivity potentiometer *R*₂ has a resistance of 1,000 ohms and allows any desired fraction of the current flowing through the electrode system to be passed through the meter, which is connected to terminals *G*. For use with the electrodes described below, a 0.1 milliampere moving coil meter is admirable. Such instruments are readily available from suppliers of radio components, in some cases quite cheaply as Services surplus. To enable the alternating current tapped off from *R*₂ to be indicated by the meter, an "instrument

1 Treadwell, W. D., and Janett, S., *Helv. Chim. Acta*, 1923, **6**, 734; Treadwell, W. D., and Paoloni, C., *ibid.*, 1925, **8**, 89.
2 Callan, T., and Horrabin, S., *J. Soc. Chem. Ind.*, 1928, **47**, 329.
3 For general references, see Glasstone, S., *Ann. Reports Chem. Soc.*, 1933, **30**, 295. For examples of more recent usage, see McElhinney, T. B., Whittemore, E. R., and Lynch, D. F. J., *Paper Trade J.*, 1938, **106**, No. 10, 37; Whittemore, E. R., Aronovsky, S. I., and Lynch, D. F. J., *ibid.*, 1939, **106**, No. 17, 33; Buras, E. M., Jr., and Reid, J. D., *Ind. Eng. Chem., Anal. Ed.*, 1944, **16**, 591; Bell, A. E., *School Science Review*, 1948, **30**, 32.

rectifier" (miniature metal rectifier) is connected as shown. As the readings of the meter are used relatively and not absolutely, the exact characteristics of the rectifier need not be known and the only marking on the dial of R_2 is an "increase sensitivity" sign.

Electrode System

Where permissible, the replacement of platinum sheet by wire in the construction of electrodes has much to commend it. Construction is usually simplified, the saving in cost is obvious, platinisation is rapidly carried out and, because of the small surface exposed to the solution, adsorption effects are minimised. The cell devised by Treadwell and Janett¹ consists of a glass cylinder closed at the lower end by a rubber stopper. The latter carries two glass tubes through the ends of which are sealed platinum wire electrodes. Rubber in contact with the solution was avoided by Callan and Horrabin,² who developed the one-piece cell shown in Fig. 2. This cell has a total capacity of 100 ml., but could, of course, be sealed down for work with small volumes.

During 1946, the author developed the micro-dipping electrode system⁴ shown at (a) in Fig. 3. The stem, which has a figure-eight section, is made by placing parallel to one another two pieces of glass tubing and fusing them together for a length of about 2 cm. Without allowing to cool, the tubes are softened all round the jointed region and are pulled out so that each bore of the double tubing is about 2 mm. When cold, this narrow portion is cut out, and forms the stem. Electrodes of 24–28 S.W.G. platinum wire are then sealed in as shown. The whole assembly is rendered rigid by fusing a bead of glass across the extremities of the electrodes. Globules of mercury in contact with the inner ends of the electrodes serve to make electrical contact with the connecting wires, which are thrust down the bores of the stem.

Recently, the construction of these electrodes has been modified as shown at (b) in Fig. 3. The somewhat tricky job of making double-bore tubing is thereby eliminated, the sealing-in of electrodes facilitated, and the construction time reduced to about 15 minutes. Two melting-point tubes *AA* of equal length are used. A length of straight platinum wire is thrust down each and sealed in at one end so that about 10 mm. or so projects beyond the seals. Leaving a few millimetres for attachment of the copper connecting leads, the other ends of the platinum wires are snipped off fairly close to the glass. The two tubes are then placed side-by-side and thus firmly retained by slipping around them two short lengths of cycle valve rubber tubing *BB*. As before, the assembly is completed by fusing glass bead *C* across the extremities of the projecting electrodes. For use when the depth of the solution is small, the electrodes may be bent as (c).

After cleaning in chromic acid and thorough rinsing,

the electrodes are lightly coated with platinum black.⁵ The coating is a rapid process, blackening commencing within a few seconds of switching on the current. A total time of 1–2 minutes, reversing the direction of the current every 10 seconds, is ample. After thorough rinsing, the last traces of the platinising solution are removed by further electrolysis for about 5 minutes in dilute sulphuric acid, periodically reversing the current as before.

Polarisation Effects

In all measurements of electrolytic conductivity, accuracy can be obtained only if polarisation is reduced to negligible proportions. If this phenomenon is at all appreciable, the point of minimum sound cannot be located precisely when attempting to find the conductivity by the usual Kohlrausch technique.⁶ In conductometric titration with direct-reading apparatus, polarisation results in reduced and variable sensitivity to changes in the conducting power of the solution brought about by added reagents. Distortion of the titration curve and erroneous or non-precise results are therefore obtained. As the current flow per unit of electrode area increases, so does the polarisation; White⁷ has drawn attention to the importance of this fact when the electrode area is small.

Kohlrausch and Holborn⁸ investigated the relationship between the actual resistance offered by the solution in the cell and the electrode area. They obtained satisfactory sound minima with resistances as low as $15/n$, where n is the area of one electrode in square centimetres, although the polarisation effects may not be negligible if this figure is less than $50/n$. It is not easy to correlate the dimensions of cylindrical-section wire-type electrodes with the parallel plates of a Kohlrausch cell, but perhaps two-thirds of the surface area of one of the platinised wires might be taken as a reasonable "effective area." For a 10-mm. length of No. 28 S.W.G. wire, n then has the value 0.079. The resistance offered by the solution must therefore be not less than about 200 ohms to comply with Kohlrausch and Holborn's condition for satisfactory audible detection, and should preferably be at least three times greater. (Further, the low frequency of the mains supply will accentuate the polarisation, so that this estimate may be on the low side).

Using a Baldwin electronic visual A.C. Null-point Detector and a Pye Kohlrausch Bridge fed at 1,000 cycles per second by a Marconi Audio Oscillator, the resistance of the electrode system dipping into 0.01 N potassium chloride solution was found to be 580 ohms at 25°C. (This corresponds to a cell constant of about 0.82). Accordingly, difficulty is to be expected when attempts are made to titrate solutions very much stronger than 0.01 N.

This conclusion was borne out by experiment. Using full sensitivity and a voltage (here 0.35) large enough to give an initial full-scale meter reading, the titration of 0.1 N hydrochloric acid with sodium hydroxide solution approximately ten times stronger gave titration curve I, Fig. 4. The relative insensitivity of the conductivity to added sodium hydroxide, particularly in the early

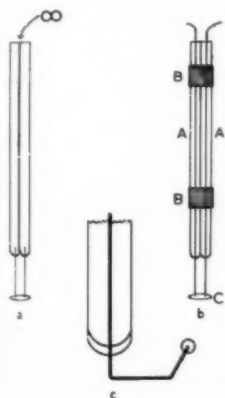


Fig. 3.—Micro-dipping electrode assemblies.

⁵ See, for example, Findlay, A., "Practical Physical Chemistry," 7th Edn., Longmans, London, 1941, p. 161.

⁶ Findlay, A., *op. cit.*, p. 157.

⁷ White, H. L., *J. Biol. Chem.*, 1933, **99**, 445.

⁸ Kohlrausch and Holborn, "Das Leitvermögen der Elektrolyte," Teubner, Leipzig, 1916, quoted by Sunderman, F. W., *J. Biol. Chem.*, 1930, **88**, 62. See also Britton, H. T. S., "Conductometric Analysis," Chapman and Hall, London, 1934, p. 19.

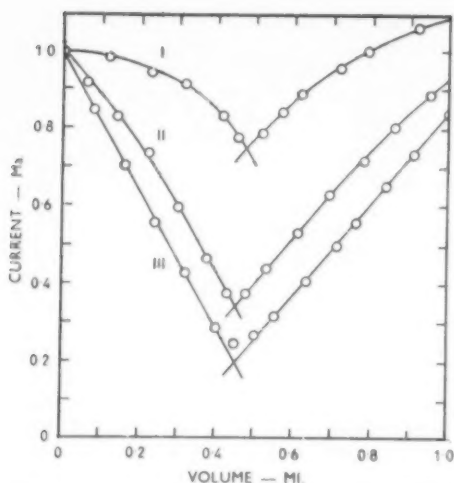


Fig. 4.—Titration of solutions of hydrochloric acid with sodium hydroxide.

stages of the titration, results in a very high "residual current" at the end point. Another effect is the pronounced departure from linearity of the arms of the curve, even though the meter readings, as in all experiments described herein, were corrected for the diluting effect of the added reagent solution by the equation:

$$i_{corr} = i_{obs} \times \frac{V + v}{V}$$

where V and v are respectively the volumes of the sample and of the added reagent. These two effects combine to render the location of the end point a matter of considerable uncertainty. When a comparable titration of 0.01 N hydrochloric acid was performed, curve II, of form satisfactory from the practical point of view, was obtained. The departure from linearity of the initial and final portions of the curve is, however, indicative of some polarisation in these regions. No such effect was noted in the titration of 0.001 N hydrochloric acid (curve III).

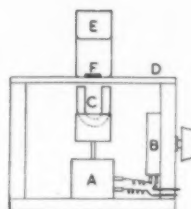


Fig. 5.—"Magnetic bar" stirrer for microchemical titrations.

Methods of Stirring

Efficient stirring is, of course, essential for rapid attainment of equilibrium conductivity during the titration. In any form of micro-electrometric titration, the presence of the electrode-system and burette-tip (plus a salt-bridge, when potentiometry is involved!) effectively fills the mouth of the titration vessel. The insertion of a stirrer of normal form is then rendered difficult. For work not involving volatile substances, a stream of purified air or nitrogen may, of course, be used, provided that the solutions are sufficiently concentrated to render negligible the effects of dissolved gases (e.g., the sweeping-out of dissolved carbon dioxide, etc., thereby slightly reducing the conductivity of the sample). Alternatively, the titration vessel, and hence the solution, may be rotated, or a miniature "magnetic-bar" stirrer may be employed. Because the sample-volumes actually examined ranged from 0.5–50 ml., it was necessary to employ titration vessels of various sizes. The larger of these vessels were small beakers, the smaller being merely cut-down flat-bottomed specimen tubes. For such cases, the "magnetic-bar" type of stirrer shown in Fig. 5 is preferable.⁹ Larger versions of this form of apparatus are, of course, available commercially.¹⁰ In the present apparatus, motive power is derived from an "Electrotor" or other small motor A, driven by a 4-volt accumulator. Variable resistance B enables the speed of the motor to be adjusted. The spindle of the motor carries a small, powerful, horseshoe magnet C†, so that the poles of the latter rotate close to the underside of the opaque perspex cover D, upon which the titration vessel E is placed. A portion of a sewing needle (or a few strands of fine iron wire) forms the core of the stirrer-bar F, which is cut from a melting-point tube and closed at both ends. For very small vessels, the stirrer-bar may be less than a centimetre long; for average use, a length of from

⁹ Unpublished experiments.

¹⁰ See, for example, "Karl Fischer Titration Apparatus," Baird & Tatlock, Ltd., London; Juleit CH/1.

[†] Rev Motors, Ltd., Bolton, Lancs.

[‡] James Neill & Co. (Sheffield), Ltd., Sheffield.

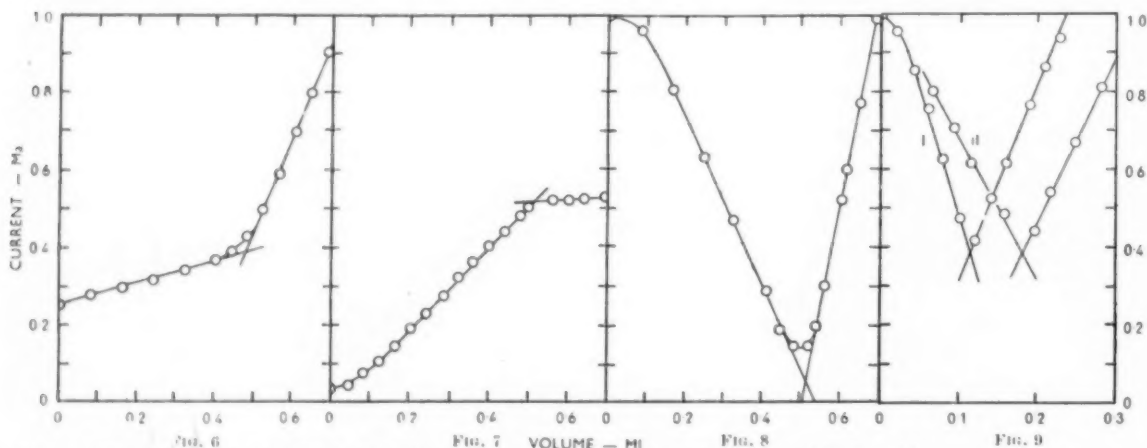


Fig. 6.—Titration of 5 ml. of 0.01 N sodium acetate solution with 0.1 N hydrochloric acid.

Fig. 7.—Titration of 5 ml. of 0.01 N acetic acid solution with 0.1 N ammonium hydroxide.

Fig. 8.—Titration of 5 ml. of 0.01 N magnesium sulphate solution with approximately 0.1 N barium hydroxide.

Fig. 9.—Titration of 0.01 N hydrochloric acid with 0.05 N sodium hydroxide. Volume of sample: Curve I, 0.6 ml.; II, 0.9 ml.

15–20 mm. is suitable. In following the motion of the magnet, the stirrer-bar rotates at the bottom of the solution, so that the agitation is greatest in this region. Vigorous stirring without fear of splashing or striking the electrodes may, therefore, be obtained. Since the speed of the motor is without noticeable effect upon the meter reading, the inductive effect of the strong magnetic field must be negligible.

Precipitation Reactions

The typical titration curves shown in Figs. 4, 6 and 7 involve fast-reacting single-phase systems. In such cases, the titration takes but a few minutes, the limiting factor being the rate of making observations. When precipitation—or other slow reactions are involved, equilibrium may not be established for some time after making an addition of reagent. It is then usually desirable, as in amperometric titration,¹¹ to allow, say, 3 minutes between addition of reagent and the taking of the meter reading. Thus in the titration of magnesium sulphate with barium hydroxide solution, the meter readings appertaining to the descending arm of the curve (Fig. 8) actually show an increase when a new portion of reagent is added.

Since the specific conductivity of a solution of a given substance is governed by the concentration of solute ions, and not by the mere number of these, there is, of course, no upper limit to the volume of sample capable of being titrated with the electrode-system described above. The straight versions (a) and (b) in Fig. 3, are suitable for volumes down to about 2 ml.; below this, the diameter of the titration vessel needs to be inconveniently small to obtain the necessary complete immersion of the electrodes. If the bent form (c) is used, it is possible to handle volumes less than 1 ml. Typical titration curves obtained in this way are shown in Fig. 9.

Although performing well with the usual run of conductometric titrations, the close spacing of the electrodes may give some trouble when flocculent precipitates are formed. This was first observed when performing titrations with solutions of certain selective organic precipitants for metals;⁹ the somewhat erratic meter readings were traced to the partial bridging of the electrode-gap by aggregates of precipitate, thus reducing the effective electrode area. In such cases, the electrode system shown in Fig. 10 may be used, when the wider spacing of the electrodes prevents bridging. The construction is mainly from glass rod and is self-evident. Connection to the wire electrodes may either be made by means of small mercury cups, as shown, or the connecting leads may be soldered to the upper ends of the electrodes.



Fig. 10.—
Electrode
system
with wider
spacing.

In the various forms of titration curve obtained by any conductometric method, the minimum conductivity is often quite a large fraction of the maximum value. With the present apparatus, this means that at no time during the process of titration does the meter show a reading of zero. For example, in the titration of 0.01 N hydrochloric acid with sodium hydroxide solution (Fig. 4, curve II), the initial full-scale deflection falls only to about one-

third. By analogy with wave-suppression in polarography by use of a counter-current device,¹² it should be possible to compensate for or balance out this "residual current." The change in conductivity could then be spread over the whole of the meter scale, thereby sharpening the angle between the arms of the curve and improving the precision and accuracy of the location of the end point.

Correspondence

THAT "FRACTURE GREMLIN"

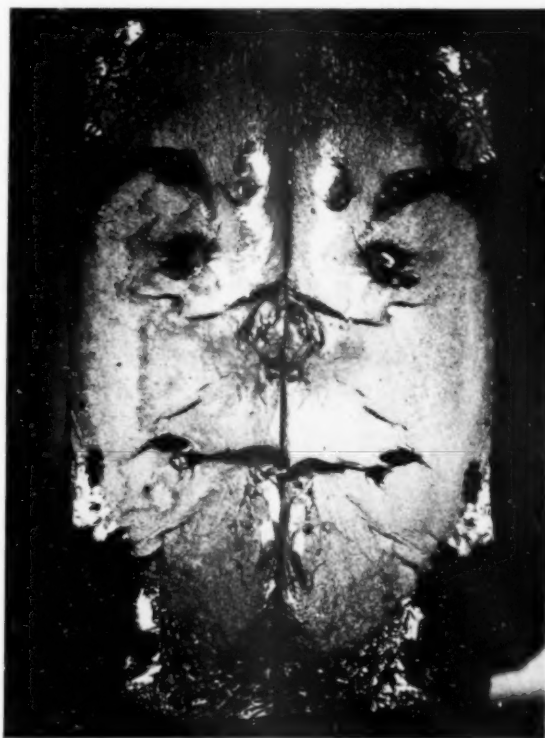
The Editor, METALLURGIA.
Sir,

While examining some photographs of corrosion-fatigue fractures, I came across this benevolent old gentleman. Surely this must be one of the few personal appearances of the "fracture gremlin." I am forwarding a print in case you think it worth reproducing.

Yours sincerely,

RALPH ERSKINE (B.Sc., A.R.T.C.)

Admiralty Road,
Rosyth.
May 19th, 1950.



The fracture gremlin appears in many guises, but there must be few who have caught the old gentleman as Mr. Erskine has done. From the interested expression it would seem that this gremlin does not dislike being photographed, as readers will note in the reproduction, and we are indebted to Mr. Erskine for sending us such an excellent print.—EDITOR.

¹¹ See, for example, Stock, J. T., in "Methods of Quantitative Micro-analysis," ed. Milton and Waters, Arnold, London, 1949, p. 475.

¹² See, for example, Stock, J. T., *op. cit.*, pp. 447, 454, 456.

A Note on Precision in Metallurgical Analysis

By Ernest H. S. van Someren, B.Sc., A.Inst.P.

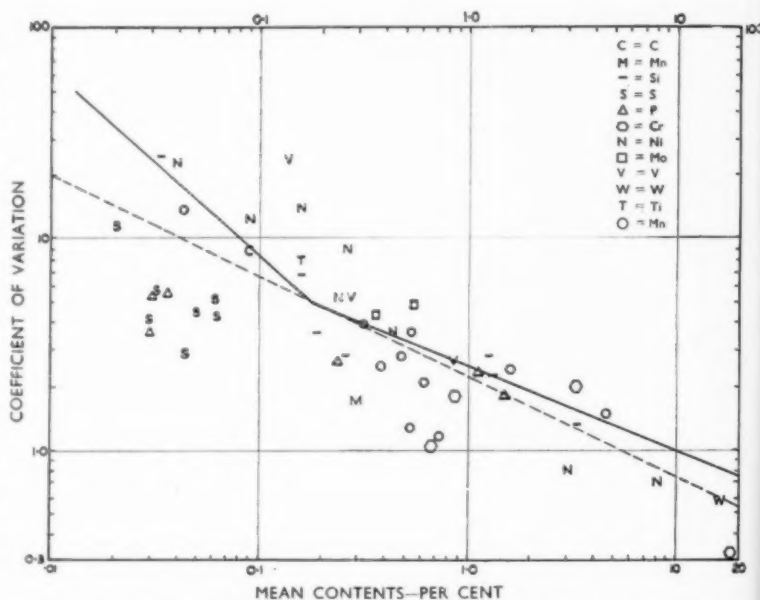
Research Physicist, Murex Welding Processes, Ltd.

A PAPER by Scheuer and Smith in this Journal (1949, 41, 44-46) dealing with the results of routine non-ferrous metallurgical analysis has suggested a typical form for the relationship between the reproducibility of estimations and the percentage concentration of the metal being determined. In order to supplement this by data on ferrous analysis a number of results have been considered—and compared with the mean curve of Fig. 1 in the earlier paper which is re-drawn as a solid line in the accompanying figure.

In this diagram the vertical ordinate (the coefficient of variation) is plotted on a logarithmic scale instead of a linear one. Our experience of routine ferrous spectrographic analysis would suggest a horizontal line at about 10% all the way from 0.04% to 20%; but since the data obtained in our own experience are complicated by the variable but definite heterogeneity of the material under test this point will not be stressed. The fact that spectrographic tests are regularly done in quadruplicate and chemical tests singly puts the chemist at a disadvantage when statistics are being collected.

One method of deriving statistics from routine work is to make use of the disagreement between duplicates where these have been done on the same sample by the same worker. Such figures can be used to derive a coefficient of variation, and this has been done for a set of analyses of carbon and manganese on mild steel wires. Carbon was determined by a visual colorimetric method and manganese by the persulphate method. Some figures kindly provided by the Chief Chemist of Murex Welding Processes (from 1942 records) gave the coefficients of variation which are denoted by the letters C and M on the figure. Since these were old records it is impossible to be sure that the statistics were fairly derived; chemists in most laboratories reject as "obviously wrong" test results in which duplicates disagree beyond some private standard. It is these rejected results which would provide the full spread of the Gaussian error function curves if the records were complete. The result for carbon falls neatly on the average line for non-ferrous metals, while that for manganese falls below it.

To supplement this, results have been selected from the whole set of figures provided for standard steels by the Bureau of Analysed Samples. Here, instead of having different tests by the same worker, we have independent tests by different workers, and often by different methods; but the increased variability from this source is compensated for by the fact that each result quoted is the average of as many tests as the chemist thought were necessary to give certainty—given



The manganese figures denoted by the letter M are drawn from records of laboratory determination whilst those denoted by small circles refer to manganese in standard steels.

the knowledge that his results were going to be published in comparison with others. Selection was necessary to eliminate groups of tests for which fewer than six figures were quoted, and the standard deviations have not been corrected for the sample size, but were converted to coefficients of variation for plotting. The tests are inserted on the same diagram as the line quoted from non-ferrous results, and it is remarkable to see that except for the figures for S and P the ferrous results fall largely around the non-ferrous line. Possibly the metallurgical importance of sulphur and phosphorous has earned them special treatment.

The ferrous results between 2 and 20%, of which there are only six, suggest that testing for nickel and tungsten is distinctly more precise than for chromium and manganese, silicon in cast iron falls intermediately.

The dotted line is the author's estimate of a mean line for the data plotted.

A recent note by Lay (*Journal Iron and Steel Inst.*, 1949, 163, 310-311) raises the question of reproducibility specifically in ferrous analysis, but since the figures quoted are ranges of an unspecified number of tests they are useless for statistical comparison.

In conclusion, it is hoped that the startling spread of the results plotted will encourage analysts to treat their own test results statistically and to publish them if they show evidence of being below average—since it is mainly by pooling evidence of our difficulties that we shall make real improvements.

The author acknowledges, with thanks, permission by the directors of Murex Welding Processes, Ltd., for the publication of this note.



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